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PRINCIPLES
OF
PHARMACY

BY

HENRY V. ARNY, Ph. G., Ph. D.

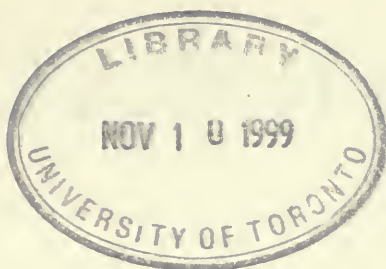
DEAN AND PROFESSOR OF PHARMACY IN THE CLEVELAND SCHOOL OF PHARMACY, DEPARTMENT OF PHARMACY, WESTERN RESERVE UNIVERSITY, CLEVELAND, OHIO

WITH 246 ORIGINAL ILLUSTRATIONS

PHILADELPHIA AND LONDON

W. B. SAUNDERS COMPANY

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AUTHORITY TO USE FOR COMMENT THE PHARMACOPŒIA OF THE UNITED STATES OF AMERICA, EIGHTH DECENNIAL REVISION, IN THIS VOLUME, HAS BEEN GRANTED BY THE BOARD OF TRUSTEES OF THE UNITED STATES PHARMACOPŒIAL CONVENTION, WHICH BOARD OF TRUSTEES IS IN NO WAY RESPONSIBLE FOR THE ACCURACY OF ANY TRANSLATIONS OF THE OFFICIAL WEIGHTS AND MEASURES, OR FOR ANY STATEMENTS AS TO STRENGTH OF OFFICIAL PREPARATIONS

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PREFACE

DURING a decade of pharmaceutical teaching, both at the Cleveland School of Pharmacy and by correspondence, the instruction given by the writer has developed into the material found in this book.

The subject of pharmacy as understood by the writer consists of the practical application of the sciences of physics and chemistry to medicines and their preparations, as well as sufficient botany to appreciate the character of those vegetable drugs entering into pharmaceutical preparations. As far as botany is concerned, the application of that science to medicine consists in that distinct study, *materia medica*, but of greater difficulty is an attempt to study the pharmacy of chemicals, both inorganic and organic, without at least some explanation of the principles underlying the science of chemistry. This explains the brief Introductory Chapters to Parts III. and IV., which students have found of value as an adjunct to their instruction in pure chemistry.

While not desiring to hamper instruction by artificial limitations, the entire subject of pharmacy is so vast that some gleaning of essentials from those substances of minor importance appeals to the teacher as imperative, and we are fortunate in having a means of elimination in the use of our national standard, the United States Pharmacopœia. Of course no pharmaceutical instruction would prove sufficient were all unofficial drugs excluded, but the existence of a pharmacopœia makes it possible to use that work as the basis of instruction, and in drilling the student in official substances ample opportunity is afforded to refer to those unofficials suggested by the pharmacopœial preparation under consideration.

The frank intention of this book is to explain the pharmacopœia from its pharmaceutical standpoint, and if that standard says that a certain chemical is "dextrogyrate ketone," or that a certain drug is a "sclerotium," the writer believes that the average student should be able to learn what such terms mean without having to search through a dozen books.

At the same time, however, the book disclaims all attempt to be a text-book in chemistry or botany, and in the bibliographical acknowledgements given at the end of this Preface there is furnished a list of complete and reliable text-books in the various sciences which go to make up pharmacy.

The book consists of seven parts:

Part I. deals with pharmaceutical processes and a striking feature is the discussion of the arithmetic of pharmacy.

Part II. deals with the galenical preparations of the pharmacopœia and those unofficials worthy of notice. It will be noticed that, wherever possible, these preparations are grouped around a typical pharmacopœial recipe, thus avoiding repetition.

- Part III. deals with the inorganic chemicals used in pharmacy, has an introductory chapter on chemical theories, and discusses chemical arithmetic very fully.
- Part IV. discusses the organic chemicals used in pharmacy, and that according to the most modern classification. Like Part III., it begins with an introductory chapter on the theory of the class of chemicals discussed.
- Part V. is devoted to chemical testing, and presents a systematic grouping of all the tests of the pharmacopœia, a feature not found in any other book.
- Part VI. takes up the prescription from the time it is written to the time it is dispensed.
- Part VII. gives the laboratory work which has been followed with much success by the students of the Cleveland School of Pharmacy. A notable feature of this part is the exercise in equation writing and in chemical arithmetic that the work affords.

Acknowledgements:

The thanks of the writer are due to Mr. J. L. Stingel, Ph.C., and Mr. O. E. Muhlan, Ph.C., for the sketches from which were made the line engravings used as illustrations. These two gentlemen, with Mr. T. M. Pratt, Ph.C., also rendered valuable service in reading proof.

The author is also very grateful to the several firms who kindly furnished cuts of their apparatus shown in the text, where due credit is given. Special mention must, however, be made of the kindness of Messrs. Eimer & Amend, of New York; and Whitall Tatum Company, of Philadelphia, who furnished for the book a large number of cuts not bearing their names. The same is true of Remington Mfg. Co., Philadelphia; George D. Feidt & Co., Philadelphia; C. H. Stoelting Company, Chicago; Boomer & Boschert Press Co., Syracuse, New York; J. M. Grosvenor Co., Boston; and Parke Davis & Co., Detroit.

Bibliographical Acknowledgements:

It was the original intention of the writer to publish at the end of each chapter a bibliography of the subjects discussed, and work in that direction was started almost as soon as the book was begun. Finding, however, that anything like a full reference to journal articles would unduly delay publication, this plan was abandoned, and in its place is given below a list of journals and works consulted, which will serve the double purpose of making acknowledgements and suggesting collateral reading to the student.

JOURNALS:

Pharmaceutical:

American Journal of Pharmacy, Pharmaceutical Review,
Druggists' Circular, American Druggist, Western Druggist,
Pharmaceutical Era, Merck's Report.

Chemical:

Berichte der deutschen chemischen Gesellschaft, Chemisches Centralblatt, Journal of the American Chemical Society, Chemical Abstracts, Journal of the Society of Chemical Industry.

In this connection special mention must be made of the facilities afforded by the Case Library of Cleveland and the Morley Library of Western Reserve University, where articles in almost every chemical journal published in English, French, and German were accessible and were consulted.

In a place by itself acknowledgement must be made to the wealth of information secured in almost every day of the writer's experience from the *Proceedings of the American Pharmaceutical Association*.

Books:

J. Q. Adams, "Report on Weights and Measures."
 Fahrenheit, Réaumur, Celsius, "Abhandlingen uber Thermometrie." (In Ostwald's Klassiker.)
 Oswald Schreiner, "History of Distillation."
 Brandel and Kremers, "The Balance"; "Percolation."
 Lommel, "Experimental Physik."
 Mann and Twiss, "Physics."
 Alexander Smith, "Inorganic Chemistry."
 Schwanert, "Lehrbuch der Pharmaceutischen Chemie."
 Roscoe and Schorlemmer, "Treatise on Chemistry."
 Thorpe, "Dictionary of Applied Chemistry."
 Bastin, "Botany."
 Prantl, "Handbuch der Botanik."
 Fluckiger and Hanbury, "Pharmacographia."
 Hatcher and Sollmann, "Materia Medica."
 Richter, "Organische Chemie."
 Sadtler, "Industrial Organic Chemistry."
 Coblentz, "Newer Remedies"; "Volumetric Analysis."
 American Med. Assn., "New and Non-official Remedies."
 Mann, "Die Moderne Parfümerie."
 Gildemeister-Hoffman-Kremers, "Volatile Oils."
 Heusler-Pond, "The Terpenes."
 Gordin, "Progress on Alkaloidal Chemistry."
 Sadtler and Coblentz, "Analytical Chemistry."
 Fresenius, "Anleitung zur chemischen Analyse."
 Hatcher, "Urinary Analysis."
 Von Jacksch-Cagney, "Chemical Diagnosis."
 Robinson, "Latin Grammar of Pharmacy and Medicine."
 Sturmer, "Pharmaceutical Latin."
 Scoville, "Art of Compounding."
 Ruddiman, "Incompatibilities in Prescriptions."

H. V. ARNY.

CLEVELAND, June, 1909.

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PRINCIPLES OF PHARMACY

PART I

PHARMACEUTIC OPERATIONS

CHAPTER I

PHARMACOPŒIAS

THE study of **pharmacy** means the study of the theories and exercise of the operations necessary to the intelligent preparing and dispensing of substances used in the healing art. The two Greek words designating this idea were *pharmacon*, "a drug, a medicine, remedy, a potion, charm, spell," and *apotheke*, "a repository; a storehouse." The first of these roots has been anglicized into such words as *pharmacy*, the art of preparing, preserving, compounding, and dispensing medicines, and *pharmacist*, one skilled in pharmacy; while from the second we get into *apothecary*, one who practises pharmacy. Another word derived from the former root is pharmacopœia: *pharmacon*, "medicine," and *poiei*, "to make"; literally, "the making of medicines," but now used to designate a book of medical recipes prepared or approved by the government of the country where published. The history of pharmacopœia means the history of pharmacy, for as pharmacopœias evolved from crude handbooks of medical lore to the present works of high scientific value, even so has the practice of pharmacy progressed from the jumbling of crude and often disgusting ingredients, of which the manipulator knew but little, to the modern blending of carefully refined constituents, of which the operator knows not only the past history, but also the future action, both chemic and therapeutic.

The earliest works answering to a pharmacopœia are Egyptian papyri, such as Papyrus No. 37 of the Berlin Museum, which is a fragment from B. C. 1700, and from its historic references seems to be a copy of a manuscript of B. C. 4000. In it five forms of medicine—ointments, liniments, poultices, clysters, and potions—are mentioned. The famous Eber Papyrus, while of later date (B. C. 1552), is peculiarly valuable because of its completeness. In it mention is made of such drugs as calamus and myrrh, salt and sodium carbonate, while its list of preparations includes all given in Papyrus No. 37, among others, pills, mixtures, and decoctions.

Passing down to classic times, we find many medical works written by individuals which may be classed among the pharmacopœias, such as books of Hippocrates (B. C. 460–377), Dioscorides (either first or

second century of the Christian era), and Galen (A. D. 131–201). These books were considered as standards by physicians for many centuries, scarcely being replaced until the appearance of the works of Paracelsus and of other medieval writers. Among the medieval works of medicine worthy of special note mention should be made of the “Ricettario,” published by the College of Medicine of Florence (1498), and the “Pharmacorum Omnium” of Valerius Cordus (1546). The latter work, published by the city of Nuremburg after the death of the writer, began a series of municipal pharmacopœias, such as those of London (1618), Paris (1639), Edinburgh (1699), which answered the requirements of the age of municipal isolation, but as means of transportation improved and people traveled freely to and fro, it was found that the difference in pharmacopœias was productive of confusion; that, for instance, a traveler from Nuremburg having his prescription compounded in Frankfort was in danger of an excessive dose of a potent preparation, there being no uniformity in the preparations of the different municipal pharmacopœias. Accordingly, a demand for national pharmacopœias grew, and the first to appear was the Codex Medicamentarius, published by the French government in 1818, the successor of the Paris Pharmacopœia, with its authority extended through all of France.

The next national pharmacopœia was that of the United States, published in 1821. The merging of the pharmacopœias of London, Edinburgh, and Dublin into the British Pharmacopœia did not occur until 1864, while in Germany the municipal pharmacopœias were replaced first by those of the States of Germany (Prussia, Hanover, Saxony, etc.), and these merged into the national authority in 1872.

At the present time there are some twenty national pharmacopœias in force, including those of every country of Europe (save some of the Balkan States and Turkey), the United States, Chili, and Mexico representing Latin America, and Japan representing Asia. Canada and other colonies of England use the British Pharmacopœia.

Even as the municipal pharmacopœias of preceding centuries caused confusion as the means of communication improved, to-day, when it is easier to travel from New York to Berlin than it was to go from Munich to Berlin a hundred years since, we are confronted by the confusion of the several national pharmacopœias. Thus every year thousands of Americans travel abroad, taking with them the prescriptions of their American physicians, and if the prescription calls for a tincture, it is quite likely that the tincture dispensed in good faith by the French pharmacist may be of entirely different strength from the American preparation, because made according to the French Codex. A most startling example of this kind is afforded by that potent preparation, tincture of aconite root. The United States Pharmacopœia of 1890 directed that 100 Cc. represent the active strength of 35 Gm. of drug (35 per cent.); the tincture of the German Pharmacopœia is but 10 per cent.; the French Codex directs it to be 20 per cent., while the strength of the British is 12 per cent. It is self-evident what dangerous results might ensue if an American pharmacist should dispense the tincture of the United States Pharmacopœia on a prescription written by a German physician; hence it is the duty of every druggist to acquaint himself with these diversities in strength, which he can easily do by reference to the two dispensatories—the United States and the National.

This diversity of strength has called forth a demand for an *international pharmacopœia*, including at least preparations of all the potent drugs, so that one strength—say of tincture of aconite—be authorized by all the national pharmacopœias, and thus the same preparation be dispensed at all pharmacies in the world. This question was discussed at the first international pharmaceutic conference held in Brunswick in 1865, and has been brought up at every subsequent conference (Paris, 1867; Vienna, 1869; St. Petersburg, 1874; London, 1881; Brussels, 1885; Chicago, 1893; Brussels, 1897; Paris, 1900), but little progress in that direction has been accomplished. One of the first practical moves beyond the stereotyped resolutions and preliminary drafts was the action in 1893 of the American Pharmaceutical Association in donating \$1000 to the international conference for defraying the expenses of compiling, publishing, and distributing such a work, and, lastly, in 1902 a conference of delegates appointed by the States of Europe and by the United States met in Brussels and decided upon a list of forty-three drugs and preparations which they recommended to the pharmacopœial commissions of all the countries of the world for adoption in the next edition of the pharmacopœia of each country.

Thus, among others, was the recommendation that tincture of aconite and tincture of other potent remedies be placed at a uniform strength of 10 per cent. in all the pharmacopœias of the world. Another recommendation was that syrup of ferrous iodide be made so as to contain 5 per cent. of ferrous iodide.

The committee preparing the new pharmacopœia of the United States embodied in that standard most of the forty-three recommendations, and the same step has just been taken by those intrusted with the preparation of the Swiss Pharmacopœia, and it is confidently hoped that all other national pharmacopœial commissions will do likewise, thus making preparations of potent remedies the same strength the world over.

Turning to our own standard, the *Pharmacopœia of the United States*, this important work was inaugurated by Dr. Lyman Spaulding of New York, who in 1817 started the movement by suggesting that the country be divided into four districts, and all medical colleges and medical societies in each district be invited to send delegates to a district convention which should form an outline of a pharmacopœia. Each convention, in turn, should send delegates to a national convention which was, indeed, held at the capitol in Washington on January 1, 1820. At this convention two drafts of prospective pharmacopœias were submitted, and these were compared, consolidated, and revised in convention and ordered published, and in the following December there appeared the first pharmacopœia of the United States. The convention of 1820 arranged that a revision should occur in 1830, by ordering the four districts to elect and send three delegates each to a convention to be held in Washington on January 1, 1830. The method of election of these delegates resulted in a misunderstanding and the holding of two conventions and publishing of two pharmacopœias; the officers of the convention of 1820 holding a poorly attended meeting at New York. The discontents held a convention at Washington, at which the surgeon of the army and the senior surgeon of the navy were present, thus giving indirect governmental authority to the pharma-

copœia so issued. Another important step was the intrusting of the publication of the revised pharmacopœia to a committee of revision of seventeen members rather than preparing a hurried work on the floor of the convention. This pharmacopœia appeared in 1831.

The convention of 1840 was arranged by the Washington convention of 1830 on lines followed to this day. Every incorporated medical society and medical college was invited to send three delegates to the convention, which met the first Monday in January.

Twenty delegates were present, and a committee of revision of seven members was elected. This committee was authorized to ask the coöperation of the pharmacy colleges in the work of revision, and, furthermore, the convention directed the presiding officer to invite the colleges of pharmacy to send delegates to the 1850 convention, which was called for the first Monday in May. The pharmacopœia was issued in 1842 and directed the process of percolation for the first time.

At the convention of 1850 pharmacists were present as delegates for the first time, and there were thirty delegates in all. A committee of four was elected to revise the pharmacopœia, and the fruit of their labors was published in 1851. The convention of 1860 was attended by thirty delegates, and a committee of revision of nine members published the pharmacopœia in 1863.

Sixty delegates were present at the convention of 1870; a revision committee of fifteen members was appointed and given general instruction as usual, and arrangements were made for the calling of the convention of 1880.

At this convention 109 delegates were present, representing thirty-five medical bodies and eleven pharmaceutic bodies. The committee of revision elected at that meeting consisted of eleven physicians and fourteen pharmacists. During the preceding decade, pharmaceutic associations have been formed in practically all the States, and accordingly the convention authorized the chairman to extend invitations to these bodies as well as to the American Pharmaceutical Association to send three delegates to the convention of 1890. Accordingly, at that convention fifty medical bodies and fifty-nine pharmaceutic bodies were represented by 163 delegates, who elected a revision committee consisting of ten physicians and sixteen pharmacists. The last pharmacopœial convention was the one held in May, 1900, at which fifty-seven medical bodies and fifty-nine pharmaceutic bodies were represented, and from the delegates present a revision committee of six physicians and nineteen pharmacists was elected. An important step taken was the perpetuation of the convention by incorporation under the laws of the District of Columbia, and intrusting the business management during the ten-year interim between conventions to a Board of Trustees which, as elected at the convention, consisted of five practical pharmacists, the president of the convention, and the chairman of the committee on revision. The next convention will be held the first Monday in May, 1910.

To a pharmacist the study of the history of his pharmacopœia should be a source of satisfaction and pride, as therein he will note the gradually increasing influence of the pharmacist in the framing of his national pharmacopœia. In 1820 the work was done by physicians only; in 1900 the pharmacists on the revision committee outnumbered

the physicians three to one. Moreover, each pharmacist should appreciate that he can have direct voice in saying who can revise his pharmacopœia, as every reputable pharmacist is eligible to membership to his State pharmaceutical association, and at the meeting prior to the convention an election of delegates to the convention is held. Lastly, the sane views of every pharmacist as to the trend of revision are given courteous attention by the committee on revision, they publishing three or four times during the decade a Handbook of Criticisms to the Pharmacopœia, compiled from articles in the pharmaceutical press or from direct communications. As mentioned in the definition on page 17, a modern pharmacopœia differs from other books on medicine by being either published by the government or by receiving governmental approval. In England and in Germany, for example, the pharmacopœia is prepared by commissions appointed by the government. In this country the national government has nothing to do with the publication of the pharmacopœia, except sending delegates from the medical staff of the army, the navy, and the marine hospital service. Moreover, the national government has but little authority in enforcing the provisions of the pharmacopœia, to each State being left the internal management of its own affairs. Hence the United States Pharmacopœia has only such legal power as the several States see fit to bestow upon it; the sole extent of authorization of the national government being its use in the several governmental departments, such as in the dispensaries of the army, navy, and marine hospital service; as the standard for the quality of the drugs entering the country through the custom-houses, and as the standard in enforcing the provisions of the National Food Law as far as drugs are concerned.

In the statutes of the several States mention is usually made of the pharmacopœia in the pharmacy and label laws, and the book is taken as the authority in interpretation of laws relating to the purity of drugs.

Any drug or medicine recognized by the pharmacopœia is said to be *official*, which term has largely replaced the term *official*, which up to ten years ago was used to convey the same idea.

THE UNITED STATES PHARMACOPŒIA AS A BOOK

Before taking up the contents of the pharmacopœia as individual drugs, it is well to devote a short space to the work as a whole, and this can best be done by copying in its entirety information found in our national standard relating to three typical officials.

In the comparative table on page 22 (*a*) is the Latin name, (*b*) the English name, (*c*) the synonym, (*d*) the official definition, and (*e*) the official description. In case of preparations, the recipe for compounding is mostly given, as (*d*), under *Tinctura Limonis Corticis*.

These several parts of the pharmacopœial reference to a drug can best be discussed in regular order.

(a)	CHIMAPHILA	SULPHUR SUBLIMATUM	TINCTURA LIMONIS CORTICIS
(b)	<i>Chimaphila</i>	Sublimed Sulphur	Tincture of Lemon Peel
(c)	[PIPSISSEWA]	[FLOWERS OF SULPHUR]	[ESSENCE OF LEMON]
(d)	The dried leaves of <i>Chimaphila umbellata</i> (Linné) Nuttall (Fam. <i>Ericaceæ</i>).	S = 31.83 It should contain not less than 99 per cent. of pure sulphur.	Lemon peel, from the fresh fruit, in thin shavings and cut into nar- row shreds, five hundred grammes 500 Gm.
(e)	Ob lanceolate, 2.5 to 5 cm. long, 8 to 18 mm. broad, the up- per portion coarsely and sharply serrate, acute or somewhat obtuse, the lower wedge-shaped and nearly entire; cori- aceous, smooth, and uniformly dark green on the upper surface, paler beneath, the veins being very prominent; odor slight; taste astrin- gent and bitter.	A fine, yellow powder, having a slightly charac- teristic odor, and a faintly acid taste. Insoluble in water; readily soluble in carbon disulphide; slightly solu- ble in absolute alcohol; more readily soluble in petroleum benzin, ben- zene, oil of turpentine, and many other oils; also in ether, chloroform, and in boiling aqueous solu- tions of alkali hydroxides. At about 115° C. (239° F.) it fuses to a yellow, mobile fluid, which upon further heating becomes dark and viscid, until a temperature above 300° C. (572° F.) is reached, when it becomes a thin liquid, boiling at 448° C. (838.4° F.). In the air it burns to sulphur diox- ide, characterized by its odor, and by its blacken- ing a strip of paper moist- ened with mercurous ni- trate T.S. held in the gas. When agitated with water, the latter gives an acid reaction with litmus paper.	To make one thousand cubic centime- ters. 1000 Cc. Macerate the lemon peel in a stoppered, wide- mouthed container, in a moderately warm place, with one thousand cubic centimeters of alcohol dur- ing forty-eight hours, with frequent agitation; then filter through puri- fied cotton, and, when the liquid has drained off completely, gradually pour on enough alcohol to make one thousand cubic centimeters of tinc- ture, and filter.
(f)	Average Dose.—2 Gm. (30 grains).	Average Dose.—4 Gm. (60 grains).	

The **Latin name** in the United States Pharmacopœia is usually the simplest and clearest title found among the several Latin designations of the drug. In this respect our pharmacopœia is a great improvement over those of most other countries, where lengthy Latin titles seem the rule. In the pharmacopœia of our country but one word is used for the Latin name whenever possible; thus, it calls aconite "*Aconitum*," and not "*Tubera Aconiti*," as does the German Pharmacopœia, there being but one part of the aconite plant recognized by the United States Pharmacopœia; hence there is no confusion in the single name. Of course, one word as the Latin name would sometimes lead to confusion;

thus, two parts of the colchicum plant are official—the root (or corm) and the seed. The single word colchicum immediately gives rise to the question, “which part of colchicum?” hence, in such cases to the Latin word is added the name of the part, viz., *Colchici Radix* and *Colchici Semen*. Again, as another case, there are two mints official—the herbs, *Mentha piperita* and *Mentha viridis* respectively. To call either *Mentha* would be confusing, hence the Latin titles of the two are as just given. In choosing the Latin name of a vegetable drug the United States Pharmacopœia usually selects the generic name (p. 25) of the plant from which it is derived, one exception being *Coca*, the plant name of which is *Erythroxylon Coca*. Among the chemicals, the same simplicity obtains in the United States Pharmacopœia, although, as it is impossible to express the idea of acids and salts in less than two words, one-word Latin names are less frequent. Lime is given the Latin name *Calx* in the United States Pharmacopœia, against *Calcaria Usta* of the German Pharmacopœia, but potassium iodide can be expressed in Latin no more simply than *Potassii Iodidum* of the United States Pharmacopœia. As in vegetable drugs, there are some cases where extra words are needed to avoid confusion. Thus, there are three official forms of ferrous sulphate—crystals, granulated, and dried. In this case the crystals are called *Ferri Sulphas*, the granulated is called *Ferri Sulphas Granulatus*, and the exsiccated is called *Ferri Sulphas Exsiccatus*. Likewise, there are two official mercury chlorides, the comparatively innocuous calomel and the very poisonous corrosive sublimate; hence it is very necessary to distinguish accurately between them. Therefore to the simple Latin phrase *Hydrargyri Chloridum* is added the qualifying adjective “*Mite*” in the case of calomel, and “*Corrosivum*” when corrosive sublimate is meant. In writing these Latin names the accepted rules of Latin grammar are followed, as will be shown later (Chapter LVII.). Here it suffices to say that all the Latin names of drugs are placed in the singular number—as *galla*, not *gallæ*, for galls; *caryophyllus*, not *caryophylli*, for cloves—this, according to the United States Pharmacopœia of 1850, “following the example of the Roman medical writers.”

English Name.—In choosing the official English name for the drug the revisers have not bound themselves to the most popular English designation for the same; in fact, as a rule, the popular names have been relegated to the class of synonyms, and in many cases as the English name has been chosen a term by which they are rarely known by the public; the official Latin and English names being very frequently the same. Thus, *Serpentaria* is given as both the Latin and English name of what is commonly known as *Virginia snakeroot*, and the latter term is used as the synonym. In choosing the English name, however, the same care and judgment were used as in the selection of the Latin name, and where the common English name is shorter than the Latin term and is sufficiently distinctive to preclude danger of confusion with other drug names, it is then chosen; thus, “cloves” is used instead of the Latin *Caryophyllus*; “ginger,” instead of *Zingiber*; “elm,” instead of *Ulmus*.

In choosing the English name of chemicals, the nomenclature of the Geneva International Conference on chemical names has been followed in many cases, the aim being to be thoroughly modern without

being ultra-advanced. Thus, while the pharmacopœia of 1880 called salts by the then accepted order (as example, "sulphate of sodium"), the United States Pharmacopœia of 1890 put itself on record by accepting the modern phraseology, "sodium sulphate." Of this nomenclature of salts, particular reference will be made in Chapter XXII, so here need only be said the advisability of the student accustoming himself to the modern naming at the outset of his studies. Thus, learn to use the modern term "potassium chlorate" instead of the antiquated title "chlorate of potassium," or, worse yet, "chlorate of potash."

Synonym.—Under this head the former pharmacopœias put those drug names too popular to be ignored, and which, for various reasons, could not wisely be accorded official preference.

The last pharmacopœia (U. S. P. Eighth Revision), however, does not include synonyms in the body of the book, these names being given in the index with reference to the official name; thus, "Snakeroot, Virginia (Serpentaria), 392," found in the index of the pharmacopœia, means that the official English name of the drug known as Virginia snakeroot is *Serpentaria*, and that this drug is discussed on p. 392 of the pharmacopœia.

The term "Virginia snakeroot" is almost universally employed instead of the official English name, "*Serpentaria*," but the pharmacopœia selects the latter word because the word snakeroot is applied to several distinct drugs, among them being: *serpentaria*; *cimicifuga*, or black snakeroot; *caulophyllum*, or Samson snakeroot; *asarum*, or Canada snakeroot; *sanicle*, or black snakeroot; *eryngo*, or button snakeroot; and *senega*, or seneka snakeroot. It is clear, therefore, that the designation snakeroot immediately raises the query, "which snakeroot?" hence the pharmacopœial aim eventually to do away with these confusing synonyms. Again, the use of the common synonyms is apt to bring misleading results by similarities in same. Thus, in one case, instead of *calamus*, recommended for the gums of a teething infant, *calomel* was purchased and applied. So the example set by the United States Pharmacopœia should be followed as far as possible by the retail pharmacist, who should endeavor gradually to educate his trade to the official English name instead of the misleading and confusing synonym.

Official Definition.—This tells what the drug is, and the United States Pharmacopœia does this in as few words as possible, as reference to the three typical cases given on the preceding page will show. According as the drug is of vegetable or of mineral origin, the definition differs; in the first case, the botanical name of the drug yielding the drug plays a leading rôle in definition, whereas with chemical drugs the symbolic chemical formula is frequently the entire definition. Hence it is profitable to discuss these two entirely different means of description separately.

Botanical Name.—Thanks to the science of botany, all the 173,706 plants known to the civilized world have been carefully studied, accurately classified, and to each has been given a distinctive scientific botanical name. This botanical name is of Latin form, and follows the rules of Latin grammar. It often seems unnecessarily long and difficult, but, as will be later shown, from time to time, it is usually aptly chosen, as by it we frequently can understand some of the char-

acteristics of the plant. The botanical name of the plant generally consists of two words—the generic name, that common to the genus or plant group, and the specific name, peculiar to the one kind of plant under consideration, an adjective usually distinguishing it from closely similar plants.

Even to the English name of a plant we frequently have to add a qualifying adjective; thus we know two kinds of oranges, and to distinguish them we call one bitter orange, the other, sweet orange; so do they in Latin, the sweet orange being *Citrus Aurantium*, the bitter, *Citrus vulgaris*. The lemon is very similar to the orange, hence is given the same generic name (*Citrus*), its specific name being *Limonum*. In fact, botanical names are very much like our personal names. We are Smiths and Jones and Browns because we belong to these families, and in order that one Smith may be distinguished from another, we call each by what is known as a given (or Christian) name—Tom, Dick, or Harry. The difference between the personal names of men and the botanical names of plants is that in plants the specific name, the plant's "Christian name," is put last instead of first, as if we would call ourselves Smith John, instead of John Smith.

Do not misconstrue the above comparison of the family name Smith to the botanical name *Citrus* into the idea that the word *Citrus* is the family name of the plant. It is the name of the *genus*; a plant *family* being much broader, and including many genera.

As to the writing of the botanical name, the generic name always begins with a capital letter, while the specific name is written with a small initial letter, except in three cases: (1) When the specific name has been at one time the generic name; (2) when derived from the name of a person; (3) when an indeclinable noun.

Following the botanical name in the United States Pharmacopœia and other standard works comes a proper name—as, "*Veronica virginica* Linné" or "*Citrus Limonum* Risso." This is the name of the botanist by whom the botanical name was bestowed upon the plants. A glance through the pharmacopœia will show that a large number of plants were examined and named by Carl Linné (1707–1778), who was a Swedish apothecary. The botanical term *family*, mentioned above, and used in the present pharmacopœia, was scarcely happily chosen, and in common conversation it is better to use the word "tribe" instead. Scientists substitute for the word the term "natural order." For complete explanation of this plant grouping recourse should be had to a standard botany, so suffice it here to state that similar plants are grouped together into a *genus* (plural, *genera*); thus all the roses are grouped into the genus *Rosa*; the oranges and the lemons into the genus *Citrus*; the mints into the genus *Mentha*. Now, roses have some characteristics like those of the blackberries (or the genus *Rubus*), and, like the apples and pears (or the genus *Pyrus*). In such cases these similar genera are grouped together for classification into a natural order; in that under consideration the name chosen for the natural order is *Rosaceæ*. Similarly, the daisies (genus *Bellis*), the sunflowers (genus *Helianthus*), the dandelions (genus *Taraxacum*), and many similar genera are grouped into one natural order called the *Composita*. It will be noticed in the United States Pharmacopœia the botanical name is always followed by the name of the natural order (or family).

Symbolic Formula.—A majority of the official chemicals are not derived from plant sources, hence in defining same a botanical name has no part, and a symbolic chemical formula is used instead.

A full explanation of a chemical formula will be given later, so sufficient for this place is the bare mention that it is an abbreviated method of expressing the composition of the chemical.

Thus the formula NaCl means a compound consisting of one atom of the element sodium and one atom of chlorine to each molecule. K_2SO_4 means a compound containing two atoms of potassium, one atom of sulphur, and four atoms of oxygen to the molecule. Hence it can be seen that the chemical formula answers in place of a definition of many words. Moreover, by means of the symbolic formula we learn the exact composition of the official chemical. Thus the name magnesium carbonate would seem to indicate the formula MgCO_3 ; that is, a body consisting of one atom of magnesium, one atom of carbon, and three atoms of oxygen to the molecule. The United States Pharmacopœia, however, puts as the symbolic formula, $\text{MgCO}_3, \text{Mg}(\text{OH})_2, 5\text{H}_2\text{O}$, showing that the official carbonate is really a mixture of the true carbonate, with magnesium hydroxide and some absorbed water. Likewise, while absolute sodium sulphate is really Na_2SO_4 , that is, it consists of two atoms of sodium, one atom of sulphur, and four atoms of oxygen to the molecule, the pharmacopœia gives as its symbolic formula, $\text{Na}_2\text{SO}_4, 7\text{H}_2\text{O}$, showing that the official product is sodium sulphate combined with seven molecules of water of crystallization. (Chapter XXVI.)

The official definitions of chemicals in the present pharmacopœia are longer than those of former pharmacopœias, because of introduction of the so-called "*Purity Rubric*." This includes a specific mention of the amount of absolute chemical contained in a commercial sample. Thus, sublimed sulphur (p. 22) "must contain not less than 99 per cent. pure sulphur."

This purity rubric was introduced to define accurately the chemical for purpose of its identification under the Food and Drug Laws, both State and national. To say that sublimed sulphur is $\text{S}=31.93$, would imply a 100 per cent. product, which is practically impossible to obtain. Since the rubric directs "not less than 99 per cent.," 1 per cent. of accidental impurity, say, moisture, is permissible.

Official Description.—This division of the pharmacopœial treatment of official drugs scarcely needs extended comment, so clear and concise are the descriptions. They are inserted to enable the pharmacist to find out whether the drugs and chemicals he purchases are really of quality worthy of administering as medicine. With vegetable drugs, the description deals with the external and microscopic appearance, and in cases of some of the more potent drugs, a method of estimating the quantity of the active ingredients is given, this method being called the *assay of the drug*. With chemicals, the official description deals with appearance, solubility, and other physical characteristics, and is supplemented by a series of chemical tests, first, to establish the identity of the chemical, and, secondly, to detect the presence of impurities. Lastly, there is usually given a quantitative assay of the chemical by means of volumetric analysis.

The neglect of the average pharmacist to compare his stock of medicines with the standards established by this pharmacopœial

description is not only unwise, but also in many States unsafe, for, aside from the ethical point that the pharmacist should never dispense a drug unless he personally knows that it is up to all pharmacopœial requirements, there is the selfish ground that in most of the older States laws relating to the purity of drugs are in force and nothing is more fatal to the reputation of a druggist than prosecution for selling impure drugs.

Method of Preparation.—Of the 958 medicines recognized by the United States Pharmacopœia, in the case of over half the method of preparation is given, the policy being to give a recipe whenever it is practical for the pharmacist to compound the remedy. For heavy chemicals, which can be made much more cheaply by the large manufacturer than by the retailer, no recipe is given, and as the pharmacopœial committee has carefully considered this question, it becomes every self-respecting pharmacist to make as many of the preparations for which a recipe is given by the United States Pharmacopœia as he possibly can.

The recipes of the United States Pharmacopœia are the result of careful study, and are as near perfect as human ingenuity can suggest. Occasionally an unpractical recipe slips in, but in most cases where a recipe yields an unsatisfactory product the fault lies with the manipulator, rather than with the framer of the recipe. In all recipes of the United States Pharmacopœia of 1900 the metric system of weight is directed to be used, the transition from the old system occurring in the revisions of 1880 and 1890, as can be noted by comparing the change in weights and measures in the pharmacopœias of 1870, 1880, and 1890:

1870. Troy ounce and fluidounce.

1880. Parts by weight (both solids and liquid weighed). This was done because of the strong desire to change from the old system—troy ounces and fluidounces—to the metric system.

However, the opposition to the innovation proved too strong, and, as a compromise, parts by weight were chosen. According to this, a preparation could be made either in avoirdupois ounces, troy ounces, avoirdupois pounds, troy pounds, grammes, or kilogrammes. Thus, in the formula of Ammonia Liniment (1880) which read—

Water of ammonia.....	30 parts
Cotton-seed oil.....	70 "

if we used ammonia water, 30 tr. oz., 30 av. oz., or 30 Gm., we would add respectively of oil, 70 tr. oz., 70 av. oz., or 70 Gm., giving everybody a chance to use his pet system of weights.

It proved, however, a bunglesome piece of work, for it was an awkward matter to weigh, for instance, 100 Gm. of a percolating tincture, but it accomplished its work. It established the idea of decimal ratio, and 1890 found the convention ready to adopt the metric system.

1890. The metric system—grammes and cubic centimeters.

DISPENSATORIES

A pharmacopœia, being intended as a concise standard work of reference, by no means embraces all the material used in the healing art. Thus the United States Pharmacopœia of 1900 recognizes 958

drugs and chemicals, and these include scarcely a tenth of the remedies that have been suggested even in our country, exclusive, of course, of the nostrums. The officials represent those remedies which, in the judgment of the revision committee, are indispensable in the practice of medicine: those remedies singled out from the vast number as being really valuable. Even of these officials the barest details are given by the United States Pharmacopœia in the effort to prevent the work from becoming too voluminous. Accordingly, in the various civilized countries there early sprang up the need of commentaries on the pharmacopœia: large works which not only fully described the official remedies, but also made mention of a large number of the unofficial drugs and chemicals used in medicine.

In this country such commentaries are called *dispensatories*, and we are provided with three valuable works of this character: The United States Dispensatory, first published in 1833, the National Dispensatory, first published in 1879, and King's American Dispensatory, first published in 1854.

At least two of these works should be found in every drug-store.

Supplemental to the recipes of the United States Pharmacopœia a book of unofficial formulas is of great value. Of these, none is better than the National Formulary, published by the American Pharmaceutical Association, and representing the contributions of the best pharmacists of America. This work has recently been given legal approval by the statement, in the National Pure Food Law, that drugs shall conform either to the pharmacopœia or to the National Formulary.

CHAPTER II

METROLOGY

METROLOGY is the science of weights and measures.

The definition of *weight* as commonly used, to express the quantity of a given substance, is erroneous, the correct word for the absolute quantity of matter in a given substance being *mass*.

Thus the mass of a substance is unchangeable under all circumstances, while the weight varies under certain influences, since weight is the measure of the gravitating force of the earth, and the gravitating force of the earth varies at different points on the earth's surface and interior.

Details of gravity can be found in any elementary physics, so suffice it to say that all substances attract other substances in direct proportion to their mass, and since of all bodies on the surface of the earth the mass of the earth is infinitely greater, the preëminent attraction is that of the earth.

That the earth attracts bodies to it is shown by every falling object, the tendency of the falling object being to attain as near the center of the earth as possible. An ocular demonstration of the quantity of gravitating force is offered by the Jolly spiral balance (Fig. 1), which

consists of an exceedingly sensitive steel spring, which is elongated on placing at the end thereof a weight which is attracted downward by the earth's force. The application of this spiral balance is seen in the ordinary spiral butcher's scale or ice scale. Such a balance measures directly the force of gravitation, and hence the weight of the substance, since weight is the measure of the gravitating force of the earth.

The same spiral balance can be used for measuring the comparative weights of various substances at the same place on the earth's surface, but since the measure of gravitation varies at various points on the surface of the earth, such balances are intended for use only at the place for which they are standardized. Thus, a body which is attracted toward the earth with a force of 192 pounds at the poles, is attracted with about a force of 191 pounds at the equator. Hence, as stated above, the weight of a substance is not an absolute criterion of its quantity, and we should learn to speak of mass rather than of weight.

In measuring the quantity of substance, three systems of units may be employed,—those of length, capacity, and weight,—and while these systems seem to have been introduced in the order named, yet their origin is so ancient as to be lost in the mists of prehistoric days, the Jews claiming that both weights and measures were introduced by Cain.

As to measures, the most ancient is the Egyptian cubit—the length of the forearm (B. C. 2300).

The *foot* is of Grecian origin, legend saying that Hercules' foot served as model. *Inch* is of Roman origin, being an "*uncia*," or twelfth of a foot, and to the same people we are indebted for the *pace* and the *mile* (*millia passum*). The *yard* is more modern, being the Anglo-Saxon measurement of the girth.

Of weights, the oldest specimens now extant are those excavated from the ruins of Babylon and Nineveh; one of these, a stone duck, dating from B. C. 2007, is inscribed as representing ten "*manah*." This manah was evidently the standard for all neighboring countries, mention of it being made in the Bible. The manah was divided into 60 shekels, and weighed, in some cases, about one avoirdupois pound (489 to 502 Gm.), while others weigh exactly twice as much.

This Babylonian manah was adopted by the Grecians, undergoing variations in weight during the lapse of years. Thus we find the city of Athens had a manah (or "*mna*," as they called it) weighing $15\frac{1}{2}$ avoirdupois ounces (436 Gm.), and the same people had a silver coin weighing $\frac{1}{16}$ of the manah, which they called drachma.

The weight standards of ancient Italy seemed also based on the Babylonian manah, the first coins from Rome being masses of bronze weighing about one-third the heavy manah, and called the "*as libralis*," or "*pondo libralis*." This Roman pound was divided into halves, thirds, quarters, and twelfths, and the latter was called "*uncia*"—hence our word "ounce." Of the same monetary value of the bronze coin "*as libralis*" was a small silver coin weighing about twenty of our grains (1.137 Gm.), and this was called a "*scripulum*," and as time went on these coin weights became the standard weights of the nation, and so it was that at the time

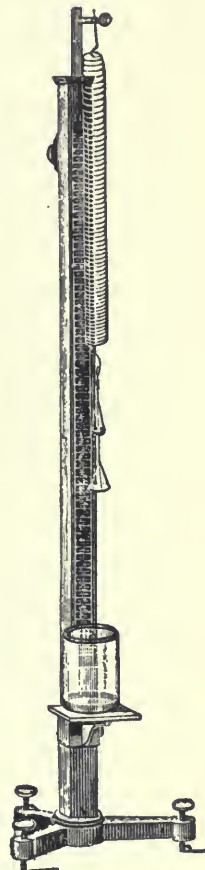


Fig. 1.—Jolly's spiral balance.

of Nero the Roman weights were a *libra*, or pound of 327 Gm.; $\frac{1}{12}$ *libra*, or *uncia*; $\frac{1}{2}$ *uncia*, or *drachma*; and $\frac{1}{3}$ *drachma*, or *scripula*, practically our present apothecary system.

Concerning weights smaller than the *scripula* there is much confusion, different authorities mentioning the *obulus*, or $\frac{1}{2}$ scruple; the *siliqua*, or $\frac{1}{3}$ *obulus*, and the *chalkus*, or $\frac{1}{18}$ *obulus*, or $\frac{1}{3750}$ pound. This is of no special interest to us, and is mentioned merely to note the absence of the grain weight, although the *siliqua* (like our grain) was the weight of a seed—that of the plant *Cerastion siliqua*, or St. John's bread.

The Roman weights were borrowed by all the countries of central Europe, and likewise we find the coins of the middle ages were copies of those of Constantine the Great, whose Eastern Empire was a direct offshoot of the Roman. Thus the earliest Anglo-Saxon penny was an exact copy of Constantine's "tremisse," and weighs $\frac{1}{3}$ *scripula*, or 24 grains, and the same is true of the penny of Alfred the Great. Evidently, this coin began to vary in weight, and to standardize same by some familiar unit was the aim of Henry III., whose statute of 1266 says the penny "shall weigh 32 wheat corns in the midst of the ear, and 12 pence do make an ounce, and 12 ounces one pound, and 8 pounds do make a gallon of wine, and 8 gallons of wine do make a London bushel." This famous statute is our formal introduction to our present system of weights and measures, giving us the first authentic reference to the pennyweight. The pound here mentioned is the ancient Tower pound, used from Anglo-Saxon times, and weighing but 5400 of our present grains.

The ordinance of Edward I. (1304) reiterates the statute just given, directing a penny of 32 grains of wheat, the ounce of 20 pence, the pound of 12 ounces, the gallon of 8 pounds of wine, and the bushel of 8 gallons. It also contains the innovation that a pound of money and of medicine consist of only 20 shillings; a pound of all other things consists of 25 shillings. It furthermore adds that a pound of money contains 12 ounces, while a pound of all other things contains 16 ounces; that is, that articles of a common character should be dispensed with more lavish hand than those which were more expensive. Following this idea, this new system of weights for the coarser commodities was called "avoirdupois," from the French words "*avoir*" and "*pois*," which mean "to have weight." Hence the origin of two different pounds and two different ounces, thus entailing upon English-speaking people even to this day material inconvenience and discomfort.

But whence the change from the penny of 32 wheat grains to the penny of 24 of our present grains? No statute authorizing a penny of 24 wheat grains can be found, unless we accept the testimony of a single and somewhat questionable authority, that a statute of Edward I. of the year 1280 directed a penny of 24 grains. The same authority believed that our present grain is the weight of a barley corn, rather than a wheat grain, and this may be the truth, since the grain of wheat of modern times has been found to weigh only three-quarters of our modern grain, and likewise only three-quarters the weight of a barley-corn.

Be this as it may, it seems possible that our modern grain is a foreign weight replacing the wheat grain, just as our two pounds (troy and avoirdupois) are foreign units which gradually forced the ancient English standard, the Tower pound, from the field.

The first official record we have of the introduction of foreign systems is in a statute of 1314, where the phrase "*pur la libre de troy orre*" is used, this ordinance directing the price of metals by the troy pound; while a statute of 1335 authorized merchants to buy and sell "*corn, wine, avoirdupois, flesh, fish,*" etc.; the word in this case has its original meaning of "*weight articles,*" and it was not until a statute of 1529 that mention of "*the weight called haverdupois*" is made.

Of these two systems, the troy is supposed to have been the standard of the French city of Troyes, which was quite a commercial center in medieval days, while our present avoirdupois pound is said to have been the standard of Bayonne, another city of France.

The United States standard yard and troy pound date from 1760, they having been conferred upon the colonies by the mother country, and are copies of the ancient weights given above, and no change has been made by this government since its organization.

In 1836 the British government modified its system of weights and measures, adopting the so-called Imperial system, in which an effort has been made to place the accepted system of weights and measures on a scientific basis by comparing same with physical units which can be easily verified. Thus, the yard of 36 inches is based on the length of a pendulum beating seconds in a vacuum in London at 62° F. The length of this pendulum is 39.1393 of the accepted historic standard inches, and 12 of these inches are called a foot, and three feet, a yard. In other words, the Imperial inch and yard are the same lengths as the old units, but their determination has been placed on a careful physical measurement. In like manner the Imperial troy pound remains at the old standard, 5760 grains. The grains, however, have been carefully determined by the weight of a cubic inch of distilled water, the weight of which is 252.458 grains. The Imperial gallon differs from the United States gallon, being the volume of ten avoirdupois pounds of distilled water under standard conditions of temperature and barometric pressure. In other words, it is the weight of 70,000 grains of distilled water, and has the capacity of 277 cubic inches.

The volumetric subdivision of the Imperial gallon is also different from the United States gallon, it being divided, as ours, into 8 pints, but the pints are divided into 20 fluidounces instead of 16.

In our present United States system of weights and measures the troy, apothecaries', and avoirdupois ounce weights, and the yard and inch, are the same as the English, but the gallon, as the old English gallon of 1760, has a weight of 58,329.6 grains, and a capacity of 231 cubic inches.

The tables of weights and measures used in the United States are:

APOTHECARIES' WEIGHT.		ABBREVIATED.
20 grains	= 1 scruple.....	℥
3 scruples	= 1 drachm.....	ʒ
8 drachms	= 1 ounce.....	℥
12 ounces	= 1 pound.....	℔

TROY WEIGHT.		ABBREVIATED.
24 grains	= 1 pennyweight.....	dwt.
20 pennyweight	= 1 ounce.....	oz.
12 ounces	= 1 pound.....	℔

AVOIRDUPOIS WEIGHT.		ABBREVIATED.
437½ grains	= 1 ounce.....	av. oz.
16 ounces	= 1 pound.....	av. lb.

U. S. FLUID MEASURE.		ABBREVIATED.
60 minims	= 1 fluidrachm.....	℥.
8 fluidrachms	= 1 fluidounce.....	℥.
16 fluidounces	= 1 pint.....	℥.
2 pints	= 1 quart.....	qt.
4 quarts	= 1 gallon.....	Cong.

The résumé of the old system of weights and measures shows what confusion prevails. We see there are four distinct *ounces*: *avoirdupois*, 437½ grains; *apothecary* or *troy*, 480 grains; *U. S. fluid*, 455.7 grains of distilled water; *Imperial fluid*, 437.5 grains of distilled water. Likewise there are two pounds: *troy*, 5760 grains; *avoirdupois*, 7000 grains. There are also two gallons: *U. S. gallon*, 231 cubic inches (58,329.6 grains of distilled water); *Imperial gallon*, 277 cubic inches (70,000 grains of distilled water).

Particular emphasis should be laid on the distinction between the U. S. and the Imperial fluid measures, and the American pharmacist should never use an English graduate.

Another pharmaceutic detail worthy of notice is the fact that pharmacists purchase their goods by the avoirdupois and dispense them chiefly by troy system. It is, therefore, well to remember that an eighth ounce of morphine sulphate is not an eighth of a troy ounce, or 60 grains, but an eighth of an avoirdupois ounce, or 54.67 grains.

COMMON MEASURES

The following common measures, largely used in the household, are worthy of mention:

- A teaspoonful is supposed to be 1 fluidrachm.
- A dessertspoonful is supposed to be 2 fluidrachms.
- A tablespoonful is supposed to be 4 fluidrachms.
- A wineglassful is supposed to be 2 fluidounces.
- A teacupful is supposed to be 4 fluidounces.

The words "supposed to be" are well chosen, since most of the spoons and other measures vary greatly as to size.

Remember that a drop is not a minim, and a minim does not necessarily weigh a grain.

Drops vary according to the character of the liquid which is being dropped, and also according to the surface from which the liquid is dropped. As to the first, the variation in size of drops is from 44 drops of syrup of acacia, 120 drops of laudanum, and 250 drops of chloroform to the fluidrachm. The size of the drop of the same liquid will vary accordingly as the surface from which the liquid is dropped is sharp or flat, or whether the orifice from which it is dropped is large or small. Thus, water dropped from the bottom of a gallon evaporating dish measures 18 drops to the fluidrachm, whereas the same water at the same temperature dropped from a glass rod of $\frac{1}{16}$ -inch bore gave 600 drops to the fluidrachm.

THE METRIC SYSTEM

The confusion resulting from the use of units of the same name and of different values cited on preceding pages led to an effort to secure an international system of weights and measures. This movement was instituted by Prince de Talleyrand, who persuaded the French assembly to pass a bill providing for the collating of the various systems of weights and measures then in use in France, requesting the coöperation of the British government, and assigning the formation of the new system, based on a universal standard, to the French Academy of Sciences. The French Academy took up the work, and the report of same was made into a law August 1, 1793. The next year the French government sent the United States government a copy of the new weights and measures, with the request that they be adopted by the infant republic; this request was communicated to Congress by President Washington in his message, January 8, 1795. In 1821 national attention was brought to the metric system by John Quincy Adams, who strongly urged the adoption of same by Congress, but nothing was done in that direction until 1866, when an act was passed legalizing the system in the United States, and directing that the same be used in the medical department of the army and navy and the marine hospital service. In recent years further progress in this direction has been attempted by strong pressure on Congress to pass laws to make the system compulsory, as well as legal. Such a bill did pass the House of Representatives in 1896, but was laid on the table, and nothing has been done since.

The metric system was based on an absolute unit, and from that absolute unit all other units were directly or indirectly evolved. The standard unit taken was the forty-millionth part of the earth's circumference around the poles, which was called a "*meter*."

This meter was divided into tenths, hundredths, and thousandths, which were called respectively decimeter, centimeter, millimeter. Multiples of the meter were also arranged. A cube, a centimeter each way, was taken as the unit of capacity, and called a *cubic centimeter*. The weight of this cubic centimeter of distilled water at 4° C. (at which temperature water attains its greatest density) was called *gramme*, and in this way, from the primal unit of length, the unit of capacity and the unit of weight were obtained.

Of course, denominations other than these three units were essential, and in all cases such units were based on the decimal scale, the change in units of length and the weight being marked by the prefixes deci-, centi-, and milli- (Latin words meaning ten, one hundred, and one thousand), as mentioned above, and deca-, hecto-, kilo- (Greek words meaning ten, one hundred, and one thousand) for the higher units.

An examination of this table of fractions,— $\frac{1}{1000}$, $\frac{1}{100}$, and $\frac{1}{10}$,—exactly like mills, cents, dimes, and dollars, will show similarity between the metric system and our currency:

10000.	Myriagramme.		
1000.	Kilogramme.		
100.	Hectogramme.		
10.	Dekagramme.		
1.	Gramme.	Compare this to	\$1.00 (dollar).
0.10	Decigramme.	"	0.10 (dime).
0.01	Centigramme.	"	0.01 (cent).
0.001	Milligramme.	"	0.001 (mill).

The unit of volume, the cubic centimeter, being very small, a thousand of these cubic centimeters was adopted as a higher unit and was called a liter; hence, a cubic centimeter is a milliliter, or one-thousandth of a liter.

Orthography.—The original French metric terms were “metre, litre, and gramme.” These have been anglicized by the American Metric Bureau into “meter, liter, and gram.” However, there is danger that the latter word, when written, may be mistaken for “grain,” hence the United States Pharmacopœia has retained the French spelling “gramme,” though accepting the American spelling of the other two units, meter and liter.

Of metric weights, we need only consider—

Kilogramme, 1000 grammes.

Gramme.

Milligramme, $\frac{1}{1000}$ gramme (0.001 Gm.).

The prefixes hecta, deka, deci, etc., should be clearly understood, but in practical work they are not used very often.

The conversion of ordinary weights and measures into the metric or vice versa is not so hard as it first seems. All that has to be known is the troy, avoirdupois, and fluid tables and the following facts:

- I. That a gramme equals 15.5 grains, approximately.
- II. That a kilogramme equals 1000 grammes, and that a milligramme is the thousandth part of a gramme.
- III. That one fluidounce of distilled water weighs, at 60° Fahrenheit, 455.7 grains.
- IV. That a cubic centimeter of distilled water weighs one gramme.
- V. That a liter equals 1000 Cc.

With the three tables of weights and measures, given on a preceding page, and the above five facts, any ordinary problem in pharmacopœial weights and measures can be worked.

Just an example or so:

(a) How many grammes in 4 avoirdupois ounces?

Answer: 1 av. oz. = 437.5 grains.

4 av. oz. = 4×437.5 grains = 1750 grains. 1750 grains.

Remember, the denomination “grains” is the bridge across which one passes from the region of the old systems to the realm of the metric system.

1 gramme = 15.5 grains.

Therefore, in 1750 grains there will be as many grammes as 15.5 goes into 1750.

$$\begin{array}{r} 15.5)1750(112 + \\ \underline{155} \\ 200 \\ \underline{155} \\ 450 \\ \underline{310} \end{array}$$

So you see there are about 113 grammes in 4 avoirdupois ounces.

In comparing this example with the tables given by the pharmacopœia it will be found that while the answer just given is 112.9 grammes, the true equivalent is 113.4. This is due to the fact that the exact value of the gramme is 15.432 grains.

If 1750 is divided by 15.432, the quotient will be 113.4. However, 15.5 is near enough for most practical pharmaceutic purposes.

- (b) How many troy ounces in 72.5 grammes?

Answer: 1 gramme = 15.5 grains.

$$72.5 \text{ grammes} = 15.5 \times 72.5 = 1123.75 \text{ grains.}$$

$$\begin{array}{r} 72.5 \\ 15.5 \\ \hline 3625 \\ 3625 \\ 725 \\ \hline 1123.75 \end{array}$$

1123.75 grains.

We are on the bridge. Now for the other side.

$$1123.75 \quad 1 \text{ troy ounce} = 480 \text{ grains.}$$

Therefore, in 1123.75 grains there will be as many troy ounces as 480 goes into 1123.75—

$$\begin{array}{r} 480)1123.75(2.3 + \\ \underline{960} \\ 1637 \\ \underline{1440} \end{array}$$

So it is seen there are about $2\frac{3}{10}$ troy ounces in 72.5 grammes.

- (c) How many fluidounces in 120 Cc.?

Answer: 1 Cc. distilled water at 4° C. = 1 gramme.

$$1 \text{ gramme} = 15.5 \text{ grains.}$$

$$120 \text{ Cc. distilled water} = 120 \times 15.5 \text{ grains} = 1860 \text{ grains.}$$

$$\begin{array}{r} 15.5 \\ 120 \\ \hline 1860.0 \end{array}$$

Again we are down to *grains*. 1860 *grains*.

$$1 \text{ fluidounce distilled water at } 15^\circ \text{ C.} = 455.7 \text{ grains.}$$

Therefore, in 1860 grains distilled water there are as many fluidounces as 455.7 goes into 1860.

$$\begin{array}{r} 455.7)1860.00(4.0 + \text{fluidounces.} \\ \underline{1822.8} \\ 3720 \end{array}$$

Hence, in 120 Cc. there are somewhat over 4 fluidounces.

- (d) How many cubic centimeters in 1 pint?

$$1 \text{ pint} = 16 \text{ fluidounces.}$$

$$1 \text{ fluidounce distilled water} = 455.7 \text{ grains.}$$

$$16 \text{ fluidounces distilled water} = 455.7 \times 16 = 7291.2 \text{ grains.}$$

$$\begin{array}{r} 455.7 \\ 16 \\ \hline 7291.2 \text{ grains.} \end{array}$$

Here we are at the crossing! 7291.2 *grains*.

$$1 \text{ Cc. distilled water} = 15.5 \text{ grains.}$$

Therefore, in 7291.2 grains distilled water there are as many cubic centimeters as 15.5 goes into 7291.2.

$$\begin{array}{r} 15.5)7291.2(470 + \\ \underline{620} \\ 1091 \\ \underline{1085} \\ 62 \end{array}$$

So it is clear that there are about 470 Cc. in one pint.

One other equivalent besides the five already given had better be mentioned.

A gramme is 15.5 grains; what then is the value of a *grain* in the metric system? That point is of value in dosage.

The dose of potassium bromide (15–30 grains) it is easy to translate into one to two grammes. But how about strychnine sulphate (dose, $\frac{1}{60}$ – $\frac{1}{30}$ grain)?

Here is where the value of a *grain* in *milligrammes* is needed.

$$\begin{array}{rcl}
 1 \text{ gramme} & = & 1000 \text{ milligrammes.} \\
 1 \text{ gramme} & = & 15.5 \text{ grains.} \\
 1 \text{ grain} & = & \frac{1}{15.5} \text{ gramme.} \\
 \text{Therefore, } 1 \text{ grain} & = & \frac{1}{15.5} \times 1000 \text{ milligrammes.} \\
 & & 155 \overline{)10000} (64 + \\
 & & \quad 930 \\
 & & \quad \underline{700} \\
 & & \quad 620
 \end{array}$$

Accordingly, one grain equals about 65 milligrammes, and the dose of strychnine sulphate is 1 to 2 milligrammes ($\frac{1}{30} \times 65$; $\frac{2}{30} \times 65$).

WEIGHTS AND MEASURES

The instruments used in the pharmaceutic application of metrology are weights and measures, and the comparison of a given substance to the standard is performed on an instrument called a scale or balance.

The only form of scale with which the mass of a substance is estimated by actual gravity is the *spiral balance*, which consists of a delicate spring to which is attached a scale pan, the elongation of the spiral when a weight is placed in the pan being marked by the descent of an arrow attached to the lower part of the spiral. This needle moves against an index, and the elongation of the spring under influence of gravity can be measured and through this the weight of the body determined (Fig. 1).

The popular modification of the same principle is shown in the common butcher's scale and ice scale.

Pharmaceutic scales are modifications of the principle of a balance which is an application of the well-known mechanical device, the lever. The principle of the lever is very simple, consisting of a rigid bar and an oscillating point called fulcrum, with the power applied in such a way as to raise or otherwise move a weight, this principle being exemplified in the familiar illustration of a crowbar for raising heavy bodies, and acting on the well-known principle that the power applied to raise or move a weight is inversely proportional to the length of the arms of the lever; hence, the longer the arm on which the power is applied, the greater the weight it can raise, and in the same way if the fulcrum is situated exactly midway between the power and the weight, these must be exactly equal to produce a state of equilibrium. On this principle the balance operates, the same primarily consisting of a rigid bar oscillating on a central fulcrum called a knife-edge. From either end of the bar are suspended pans on which are placed respectively the power and the weight; the "power" in this case being the standard weight used in estimating the quantity of the substance. If the central knife-edge is situated exactly midway between the two, a standard weight will be balanced by exactly the same mass of substance desired placed in the other pan; if, on the other hand, the fulcrum is so placed that one arm of the beam is longer than the other, a standard weight placed in the pan suspended from the longer arm will weigh as many times the amount of substance in the pan placed on the short arm as the relative length of the arms.

For the purpose of studying pharmaceutic balances it is convenient to divide them into the following four classes:

- Single beam equal arm balances.
- Single beam unequal arm balances.
- Double beam unequal arm balances.
- Compound lever balance.

SINGLE BEAM EQUAL ARM BALANCES

Several requirements are stated by authorities as essential to a perfect prescription balance, the first being that *the point of suspension should be slightly above the center of gravity*. By the center of gravity is meant the point on which a given object will oscillate absolutely freely: that point at which the weight of an oscillating body is exactly compensated in every direction.

To place the central knife-edge exactly at this point is to make the balance so sensitive that time is lost in attempting to bring the beam into a state of rest.

To place the knife-edge below the center of gravity means that abnormal condition wherein there is a greater weight above the point of suspension than below, with the result that, at the first opportunity, the heavy part above will endeavor to invert itself.

Second, *the end knife-edges should be equidistant from the center, and all three should be on the same plane and parallel to one another*.

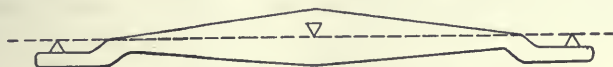


Fig. 2.—Position of knife-edges.

It is self-evident that if the beam is of exactly the same weight throughout, and an equal arm balance is desired, that condition can be obtained only by having one end knife-edge exactly the same distance from the central as the other. As the end knife-edges are usually inverted in relation to the central knife-edge, the effect of placing these knife-edges on the same plane is shown in the appended sketch (Fig. 2).

Third, *the beam should be of inflexible and light material*.

The lighter the beam, the less friction does it exert on the central knife-edge, hence a dictum in successful scale manufacturing is to make the frame as light as possible without sacrificing rigidity.

The knife-edges in ordinary prescription balances are of hard polished steel, but as that metal is liable to rust from contact with moist air, and since every particle of rust will increase the friction, rusted knife-edges oscillate less freely. In the finer forms of balance this impediment to free oscillation is prevented by making the tips of the knife-edges of agate, which does not rust.

Not desiring to fill this work with an excess of pictures which can be found in any catalogue of a manufacturer of pharmaceutic apparatus, only typical cases of the several forms of balances will be given.

Testing the Balance.—The testing of a balance to see whether the above requirements are met with is quite a simple matter. Thus, if a scale is sold as being sensitive to a certain weight, say $\frac{1}{100}$ grain,

it should show its sensitiveness when such a weight is placed on a scale alone, and also when the scale is burdened with its limit of weight.

If the arms of the balance are exactly equal, the state of equilibrium produced by putting a hundred grammes weight on either pan should remain the same if these weights are exchanged.

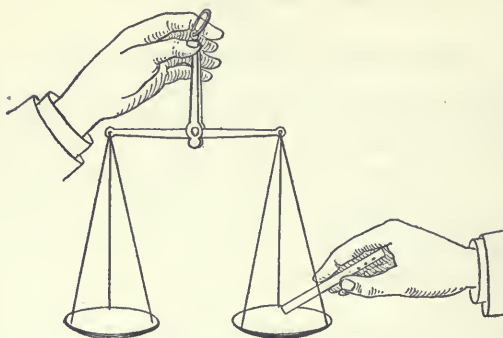


Fig. 3.—Holding hand scale.

If the knife-edges are balanced, moving the weights to various parts of the scale pans will have no effect on the equilibrium of the balance.

Hand Scales.—The simplest form of single beam equal arm balance consists of a beam with pan suspended on either end, the beam itself hanging from a ring held by the hand. In using a hand scale,

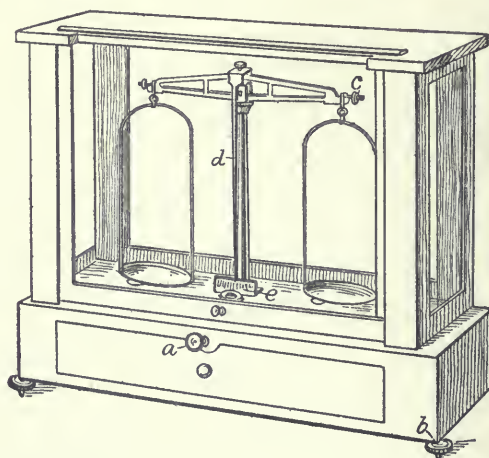


Fig. 4.—Prescription balance: *a*, Lever for raising beam; *b*, leveling screw; *c*, adjusting button; *d*, index needle; *e*, index.

the ring from which the beam is suspended should be placed on the first finger of the left hand, leaving the thumb and second finger to prevent too great oscillation of the index (Fig. 3).

Prescription Balance.—In this balance the beam is not suspended from the hand, but oscillates on a rigid upright column of brass through

the center of which passes a lever by which the beam can be raised for oscillation, and lowered in a state of rest, at which time the knife-edges are not in contact with the oscillating surface, hence useless friction is avoided. The entire apparatus is inclosed in an appropriate glass case (Fig. 4).

In purchasing a prescription balance it is highly advisable to secure one which has a spirit-level and is provided with leveling screws, since to obtain accurate results in weighing it is essential that the balance be perfectly level.

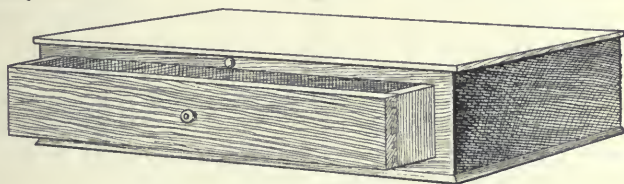


Fig. 5.—Army prescription scale (folded).

After continued use it will be found that the weight of the two arms may vary, and thus throw the balance out of equilibrium, and to remedy this, the better forms of such balances have a beam on each end of which are buttons which move freely up and down a screw, thus making it easy to attain equilibrium.

Attached to the beam near the central knife-edge is the metallic needle, which, by its oscillation against the scale, shows when equilibrium is reached. This is attained when the needle, after oscillating, comes to rest at the point on the index directly beneath the point of suspension. To wait until a delicate balance ceases oscillating is a tedious operation, and therefore the pharmacist and chemist have recourse to the method known as reading the zero point, explained under the analytic balance.

Army prescription balance is an exceedingly simple and convenient form of single beam equal arm balance, but not of sufficient accuracy to warrant general use in pharmacy. As suggested by the name, it is primarily intended for use by army surgeons and others who move from place to place, being of such manufacture that it can be easily dismantled and carried around in the drawer (Figs. 5 and 6).

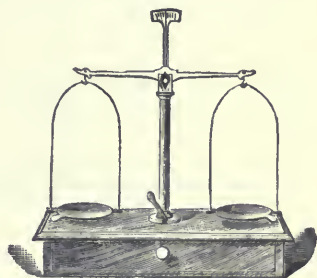


Fig. 6.—Army prescription scale.

Analytic Balance.—Merely a refined modification of the prescription balance. While the latter is sensitive to $\frac{1}{30}$ grain, the analytic balance is sensitive to $\frac{1}{20}$ milligramme, or $\frac{1}{1300}$ grain. In the analytic balance the beam is usually of aluminum, while the knife-edges are invariably tipped with agate.

For weighing a smaller quantity than a centigramme recourse is had to a rider weight. The rider, which usually weighs a centigramme, is so shaped as to straddle the beam, and therefore when placed on the beam, exactly over the end knife-edge, it will exert the weight of a centigramme. The distance on the beam from the end knife-edge to

the central knife-edge is divided exactly into a hundred parts, and up and down the beam on the scale the rider can be moved. Following

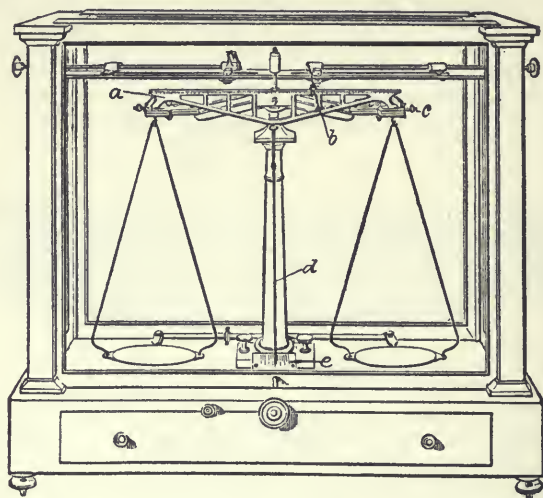


Fig. 7.—Analytic balance: *a*, Graduate beam; *b*, rider; *c*, adjusting buttons; *d*, index needle; *e*, index scale.

the principle of the lever, weights which will exert on the opposite scale pan, the force of 1 centigramme when placed exactly over the end

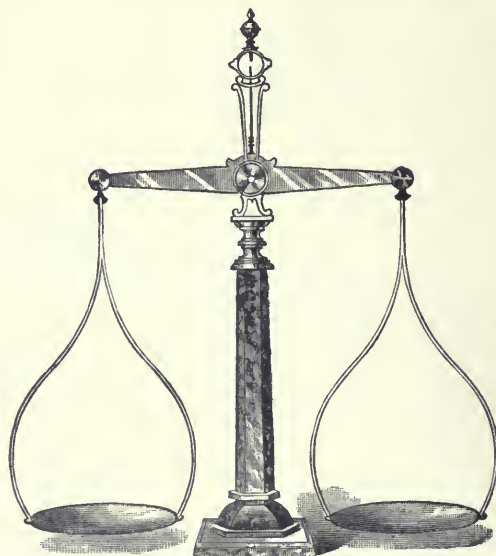


Fig. 8.—Marble counter balances.

knife-edge, will exert the force of but $\frac{1}{2}$ centigramme if moved half way up toward the central knife-edge, and will exert the force of but

$\frac{1}{10}$ centigramme if moved nine-tenths of the way up toward the central knife-edge. The beam being divided into exactly 100 equal parts, the rider can be placed at any of these marks, and thus can be obtained on the other scale pan any part of $\frac{1}{100}$ centigramme or $\frac{1}{10}$ milligramme (Fig. 7).

Reading Zero.—Time is saved in estimating equilibrium by watching the oscillation of the needle against the index mentioned under prescription balances. The index scale on the analytic balance is usually divided into twenty equal lines, ten on either side of the line exactly below the point of suspension.

We set the beam in oscillation, when the needle will move to the left, say five lines, and then, if the beam is in equilibrium, the needle will move back to exactly the fifth line on the right. On the return the needle will go to almost the fifth line to the left. If, however, the index moves to the fifth line to the left and then returns to the second line to the right, it would indicate that the right beam was heavier than the left beam, and equilibrium must be restored by moving a button at the end of that beam.

The careful estimation of weight by this means is called "interpolation," full details of which can be found in text-books on advanced physics.

A large form of single beam equal arm balance is found in the old-fashioned counter balance shown in Fig. 8.

SINGLE BEAM UNEQUAL ARM BALANCE

In this form of balance the beam, from which is suspended the weight, is usually two to ten times as long as the beam to which is attached the pan on which the substance is to be weighed. Thus the principle of action, as cited above, is that the standard weight is to the substance to be weighed as the length of the short arm is to the length of the long arm. Thus, on such a balance, in which the relative length is one to ten, a weight of one pound placed on the end of the long arm exerts a weight of ten pounds placed at the end of the short arm, and in such balance the weight is usually suspended directly from the beam, which is accurately divided into 100 parts as the beam of the analytic balance, the weight serving in this case the same purpose as the rider in the analytic balance.

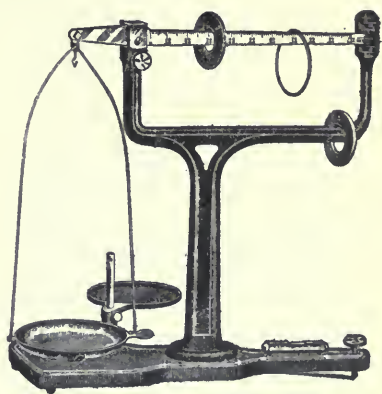


Fig. 9.—Chaslyn balance.

An interesting type of such balance is found in the Chaslyn balance (Fig. 9).

DOUBLE BEAM UNEQUAL ARM BALANCE

This balance is a modification of the preceding one, it being found convenient in many cases to have two beams instead of one, so as to

handle two weights. Such a balance is particularly convenient when

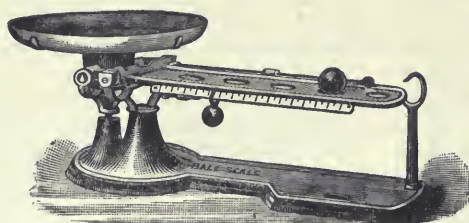


Fig. 10.—Troemner's ball scale.

it is desired to know the tare of a utensil into which the weighed substance is to be placed (Fig. 10).

COMPOUND LEVER BALANCE

In this balance we find a series of levers replacing the single or double beam hitherto discussed, one lever acting on another. The more points of contact, the more the friction, and therefore such forms

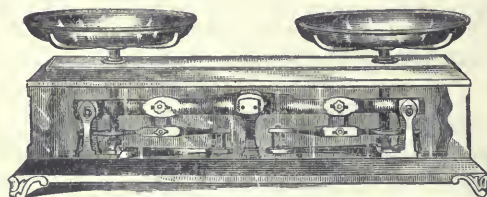


Fig. 11.—Troemner's glass box scale.

of balances are, as a rule, less accurate than the single beam; however, they are more durable, and, as a rule, weigh more quickly. Such a form of balance is seen in the usual box counter scale and the box prescription scale (Figs. 11 and 12).

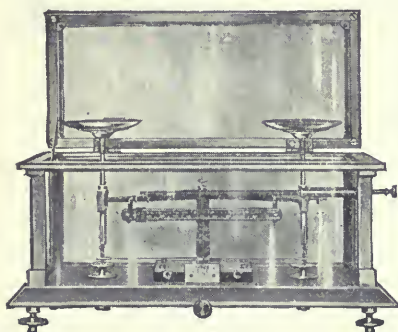


Fig. 12.—Troemner's prescription scales.

In both cases the levers are so arranged that a weight placed on a scale is exactly compensated by the same weight on the other beam, hence to all intents and purposes the principle of action is the same as that of the single beam balance.

A modification of the compound lever balance is seen in the ordinary platform scale intended for weighing very heavy articles. In such cases the handling of weights equal to the mass of substance to be weighed would entail vast labor, so such compound lever balances are constructed on the principle of unequal beam balances, consisting primarily of two levers, on each of which the power is multiplied by ten. In such a balance the weight of one pound placed on the first

lever exerts on the end of the second lever a force of ten pounds, and this force of ten pounds produces a force of 100 pounds on the end of the second lever on which the platform rests; hence on such platform scale a one-pound weight placed on the end of the index arm will exert the force of 100 pounds on the platform.

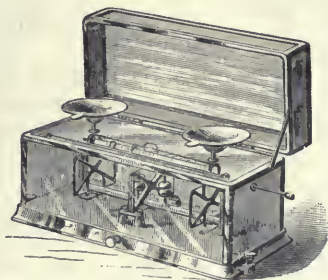


Fig. 13.—Torsion balance.

Torsion Balance.—As cited above, the chief objection to steel knife-edges is that when they rust, the friction is increased, with a corresponding loss of sensibility. This is obviated by the use of agate knife-edges, but that materially increases the cost of the apparatus (Fig. 13).

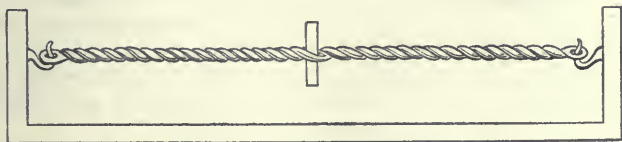


Fig. 14.—Effect of torsion.

In the torsion balance knife-edges are dispensed with and, instead, the beam oscillates on a tightly drawn and twisted wire (Fig. 14).

CARE OF THE BALANCE

In the balance we have the most accurate physical instrument that has been devised, and also one which, under reasonable treatment, will last many years. In that event, however, due care must be taken to protect the balance from injury, and among the several points which must be enumerated are these:

First, the balance should be kept permanently in a dry room, the temperature of which is as even as possible, and protected from any acid or corroding vapors. In case it is necessary to weigh a fuming liquid, such as nitric acid, it should be placed in a well-stoppered bottle while weighing.

Second, such suggestions as not placing the substance to be weighed directly on the metallic pans, and keeping the pans free from all moisture, seem unnecessary to one who appreciates the value of a balance, and convenient for accomplishing these results are the movable glass scale pans which can be purchased from any scale manufacturer.

Two other rules should be rigidly enforced by the owner of the balance.

Third, weights should never be left on the balance, and, whenever possible, should be transferred to the balance by means of pincers.

Fourth, under no circumstances should the balance be left to oscillate after weighing is finished, and during the weighing it should be brought to a state of rest whenever new weights or a new substance is being placed thereon.

WEIGHTS

The weights used on scales and balances are composed of iron, brass, or aluminum, and of such shape as convenience suggests. Thus, the cheap avoirdupois weights are made of iron, while those finer are composed of brass, and the more expensive are nickel plated, and their shapes are usually those shown in Fig. 15.

The troy weights are usually made in sets nested, each weight, therefore, being a cup inclosing all the weights of smaller denominations than itself, the smallest, usually $\frac{1}{4}$ troy ounce, being solid, and fitting in a cup-shaped weight of the same denomination (Fig. 16).



Fig. 15.—Brass block weights.



Fig. 16.—Troy cup weights (solid brass).

As the avoirdupois weights, so the metric are composed of iron or brass; fine analytic metric weights are sometimes gold-plated brass and are sometimes platinum coated.

In purchasing avoirdupois and metric weights it is usually wisest to choose such as are arranged in a block. A very convenient form of such block is made of cast iron, the objection to wooden blocks being that they frequently swell, rendering it difficult to remove the weights.

The metric analytic weights should be in a fine wooden box, the depressions in which the weights rest being lined with velvet, serving the double purpose of keeping the weights from being scratched and preventing them being held fast by the swelling of the wood. The denominations under one gramme are made of sheet platinum.

For dispensing purposes troy weights are used, the denominations under ten grains being made of aluminum, either in sheets or in the form of wire, the latter being bent into as many sides as the weights have grains.

MEASURES

In pharmacy the larger measures, gallons, quarts, etc., are not often used, although reliable measures of these sizes should be found in every well-regulated pharmacy. These are made of brass, tinned iron, or enameled iron.

Indispensable in the prescription department are graduates or measures, from a pint down, made of glass, with the subdivisions of the unit marked on the sides (Fig. 17). The graduation of these measures is performed in two ways: in one the plunger used in making the utensils out of the molten glass is graduated; thus the finished

graduate is produced in one operation. More popular, however, are those in which the graduating is done by hand, the quantity of liquid



Fig. 17.—Eight-ounce graduate.

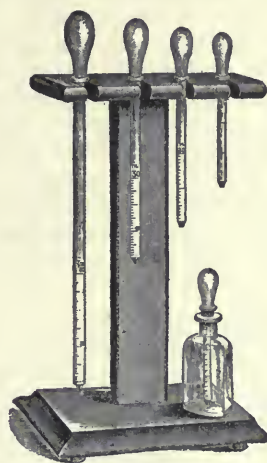


Fig. 18.—Squibb's minim pipettes.

being poured into the ungraduated vessel from a burette, and this quantity of liquid is carefully marked, the finishing of the graduate being done by the engraver.



Fig. 19.—Liter flask.



Fig. 20.—Volumetric pipette.

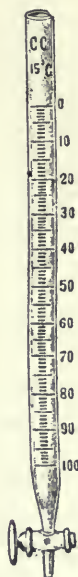


Fig. 21.—Burette.



Fig. 22.—Meniscus (Sollmann).

The subdivisions of graduations are into drachms and minims in ordinary graduates, and into cubic centimeters in the metric.

The minim graduate, however, is far from being reliable, as a large amount of the liquid is retained by capillarity of the glass vessel, and, therefore, for measuring minute quantities, a pipette should be used. Such pipettes, graduated to minims, were put on the market by Dr. E. R. Squibb (Fig. 18), and are more reliable not merely by reason of the dispensing of the full quantity of the liquid, but, being narrower than a graduate of equal capacity, there is less chance for error in pouring more or less of the liquid into the utensil; the narrower the graduate, the more accurate can the quantity desired be determined.

In this way, for delicate analytic work, a liter graduate is rarely used, there being substituted for same a flask of such size that when filled with a liter of water the water rises within the narrow neck of the flask. Such liter flask is used chiefly in volumetric analysis, liquids being delivered and measured from same by means of pipettes (Fig. 20) and by burettes (Fig. 21).

In both cases the liquid is measured within a narrow tube, hence based on the same principle as the liter bottle (Fig. 19).

In placing liquids in a comparatively small utensil, like a burette, it will be observed that, through capillarity, the liquid is attracted to the sides of the vessel, thus making an edge which it is sometimes difficult to read; such an edge is called a "meniscus," and in reading the quantity of the liquid, the line representing the quantity should be exactly in the center of the meniscus (Fig. 22).

It is hardly necessary to state that in measuring liquids in a graduate the latter should be held perfectly level and the line representing the desired quantity even with the eye (Fig. 23).



Fig. 23.—Pouring into graduate.

CHAPTER III

SPECIFIC GRAVITY

IN Chapter II care was taken to mention that 455.7 grains was the weight of a fluidounce of distilled water. Why distilled water? Would it not be the weight of a fluidounce of chloroform as well? Common sense teaches that "some liquids are heavier than others," as we put it—that chloroform is heavier than water. A fluidounce of chloroform weighs 678.9 grains, against 455.7 grains, the weight of the same volume of water. Accordingly, we see that chloroform is nearly one and one-

half times heavier than water. That gives the idea of specific gravity, the definition of which can be given as *the relative weight of equal bulks of different bodies*, adding thereto the provision that, for solids and liquids, the unit of specific gravity is distilled water.

Now, to go back to our chloroform example: the volume, the bulk, the capacity of that one-ounce graduate remains the same whether it contains water or chloroform. Therefore a fluidounce—be it of water or of chloroform—represents “equal bulk.” The respective weights of a fluidounce of the two liquids is “relative weight,” and, reducing to unity, the specific gravity is obtained.

Instead of the term specific gravity, the word “density” is the more happy choice, since the term “specific gravity” suggests the gravitating force of the earth, which has only indirect connection with the subject.

The estimation of the specific gravity of various substances is of value, first, as the indication of purity or strength of the substance, and, second, through the data thus afforded we can estimate the volume of the substance, while in a minor degree it is sometimes of service in the diagnosis of disease.

The first application of density given above can be best explained by a simple illustration, thus: The average 92 per cent. sulphuric acid has a specific gravity of 1.834; in other words, is 1.8 times heavier than water. If this acid is mixed with water, the density of the liquid will vary to somewhere between 1.834 and 1.000, and, indeed, varies in proportion to the amount of water added.

In the same way official 95 per cent. alcohol has a specific gravity of 0.820, and is, therefore, 0.82 times as heavy as water. If such alcohol be mixed with water, the specific gravity of the liquid will rise to some point between 0.820 and 1.000, and, indeed, in proportion to the amount of water added; hence by taking the specific gravity of such liquid, we can estimate the exact strength of the liquid. Tables of this character have been carefully worked out, and are found in the pharmacopœia.

The other two uses of specific gravity being of minor importance, will be discussed in the appropriate place.

Estimation of the density of a body differs according as the substance is solid or liquid, and since, by the explanation given above, the specific gravity of liquids becomes simple, we will first discuss the estimation of the specific gravity of liquids; afterward, that of solids.

There are several ways of estimation of the specific gravity of liquids: (a) By use of pycnometer or specific gravity bottle; (b) with the hydrometer; (c) with the Mohr-Westphal balance; (d) with Lovi's beads; (e) with the Jolly spiral balance.

ESTIMATION OF SPECIFIC GRAVITY OF LIQUIDS

With the Pycnometer.—As mentioned above, the estimation of the specific gravity of a liquid is rendered simple because of the ease with which the weight of a bulk of given liquid can be compared to the weight of an equal bulk of water. Thus, in the simplest methods of estimation all that is necessary is to estimate the weight of a fluidounce of water in a graduate, then pour out the water, dry the graduate,

pouring in the same measure of the liquid, the specific gravity of which is desired, weighing same, and then comparing the weight of the fluid-ounce of water with the weight of the fluidounce of liquid. As many times heavier as is the weight of the fluidounce of the liquid than the weight of the fluidounce of water, so many times heavier is that liquid than water, which figure represents the specific gravity. The inaccuracies of this method of estimation are due to the same causes as any inaccuracy in the measurement of liquids by means of the graduate, as mentioned in the preceding chapter, viz., the uncertainty in reading an exact volume of a liquid when placed in as wide a vessel as a graduate; hence, in order to obtain accurate results, we generally weigh the liquid, the weight of which is to be compared to the weight of an equal



Fig. 24.—Pyknometer.



Fig. 25.—Squibb's pyknometer.

bulk of water, in a flask called a *pyknometer*, two modifications of which are shown in Figs. 24 and 25.

The only advantage a pyknometer (or specific gravity bottle) has over the graduate method just given is its greater accuracy. By tilting a graduate in measuring it is very easy to get a little more or a little less than the quantity desired, and every grain counts in estimating specific gravity.

In glancing at the narrow neck of the pyknometer it can be seen how the danger of incorrect readings is lessened. Then, too, the pyknometers usually hold 1000 grains or 100 grammes, which renders long division unnecessary. If a 1000-grain bottle holds 1500 grains of a certain liquid, it is obvious that the specific gravity of that liquid is 1.50.

In both cases the possible variation in the volume of the liquid when manipulated by a person of ordinary intelligence is practically nil.

The method of estimating specific gravity by means of the pyknometer is by all odds the most accurate means at our disposal, but the process is one requiring considerable time. To facilitate more rapid reading other instruments have been devised, most useful of which is the *hydrometer*.

The Hydrometer.—As to the principle of a hydrometer, if asked in which fluid one could float better, in fresh or in salt water, the instant answer would be in salt water. Why? Simply because salt water, being heavier, buoys one up more. And, following the rule, the heavier the liquid, the more it will buoy a body up, and, indeed, proportionally. To prove this may be cited that while a piece of iron will sink in water, on mercury it will float around like a chip in water. Why? Simply

because while iron is eight times heavier than water, mercury is thirteen times heavier, and the lighter iron will float on the heavier mercury.

The hydrometer consists of a glass tube at the base of which two bulbs are blown, one containing enough shot or mercury to enable it to obtain an upright position when immersed in water, and the other and upper one filled with air (Fig. 26). According to the theorem of Archimedes, a body when immersed in water loses as much weight as the weight of the water it displaces, that is, a body displaces its own weight of a liquid and, accordingly as the liquid is lighter or heavier than water, so does the hydrometer sink or rise when immersed in same.

There are two classes of hydrometers, in one of which the weight of the hydrometer remains the same, whereas the point at which it floats varies accordingly as the liquid is lighter or heavier, and the other form, which is designed to float always at the same point in a liquid, which feat is accomplished by weighting down the hydrometer when immersed in a heavy liquid, *i. e.*, by meeting the buoyancy of the heavy liquid by adding weights in a pan at the top of a floating instrument.

Of the first class, we have such well-known hydrometers as Baumé's and Daniel's, while on the principle of the second type are based the Nicholson, Fahrenheit, and the Mohr specific gravity balance.

Baumé's Hydrometer.—The sketch and explanation on preceding page explain the construction of the above-named hydrometer, except details as to its graduation.

In constructing the hydrometer, it was early noted that such an instrument designed to measure accurately the specific gravity of the commonly known liquids, ranging, say, from ether (0.720) to bromine (3.000), would of necessity be so long as to be inconvenient.

Baumé, at the outset, made two forms of this hydrometer, one for liquids heavier than water, and one for liquids lighter than water. The one for heavier liquids is graduated by weighting same with sufficient shot so that it would be almost completely immersed in water, and the point which was in contact with the surface of the water was marked or denominated zero. He then immersed this instrument in a 15 per cent. solution of common salt, whereupon, by reason of the superior buoyant force of this liquid over water, the hydrometer was pushed up and the point of contact of the hydrometer with the surface of the salt solution was marked off and denominated 15, and the space between the zero mark and 15 accurately divided into 15 spaces (provided always that the circumference of the tube was the same all along its length).

The remainder of the tube was divided into spaces equal to the 15 spaces between the water contact and the solution of salt contact, and this marking was arbitrarily denominated "degrees Baumé heavy."

In a similar manner was the hydrometer for lighter liquids constructed, this hydrometer being weighted so as to float in a 10 per cent. solution of salt at a point very near the base of the tube, and this point was marked zero.



Fig. 26.—
Hydrometer
(schematic).

It was then floated in water, when it naturally sank, and the place of contact with the surface of the water was marked 10, and the spaces between the water contact and the salt contact divided into 10 equal spaces, which spacing was continued up to the length of the tube, and in turn designated "degrees Baumé light." Thus were the original Baumé hydrometers constructed, but at present they are standardized by dipping the hydrometer in water and then into a standard liquid (concentrated sulphuric acid for the "heavy" hydrometer; ether for the "light" one), marking the floating point, the "degree Baumé" corresponding to the specific gravity of the standard liquid, and then subdividing the space between the floating point in water and in the standard liquid into the number of uniform spaces equal to the difference in "degrees" between water and the standard liquid.

This description shows how exceedingly arbitrary and confusing was the Baumé marking, for not only have we "degrees" which in themselves signify nothing, but we have "degrees Baumé light" and "degrees Baumé heavy." Thus, when the statement is made that the specific gravity of the liquid is 15° Baumé, the first thing that we must find out is whether a light or a heavy hydrometer was used, and, what is more unfortunate concerning this Baumé marking, is the fact that it is practically universally used in commerce where heavy chemicals are bought and sold by degrees Baumé rather than by their actual specific gravity.

It therefore behooves every retail pharmacist to be able to convert degrees Baumé into specific gravity rapidly, which can be easily done by using the following factors:

To convert heavy degrees Baumé into specific gravity:

$$\frac{145}{145 - ^\circ \text{B.}} = \text{sp. gr.}$$

Thus 20° B. heavy =

$$\frac{145}{145 - 20} \text{ or } \frac{145}{125} \text{ or } 1.16.$$

To convert light degrees Baumé into specific gravity, use the following factors:

$$\frac{140}{130 + ^\circ \text{B.}} = \text{sp. gr.}$$

Thus 20° Baumé light =

$$\frac{140}{130 + 20} = \frac{140}{150} = 0.933.$$

Aside from these general features of the hydrometer we find modifications of the hydrometer especially adapted for taking the specific gravity of certain liquids; thus we have the *alcoholometer* for estimating the strength of alcohol through the specific gravity. Usually, the marking of this form of hydrometer is not in terms of specific gravity, but in the actual percentage of alcohol. In the same way we have the *lactometer* for estimating the strength and density of milk; the *saccharimeter* for estimating density of syrups; and the *urinometer* for estimating the density of urine.

Urine differs in specific gravity from 1.010 to 1.070, and since the instrument is very small, the marking is from 10 to 70 instead of the actual specific gravity, as stated above.

The best form of urinometer shown in Fig. 27 was devised by Dr. E. R. Squibb.

Nicholson's Hydrometer.—This apparatus is of scientific interest because its principle of action differs from that of the hydrometers



Fig. 27.—Squibb's urinometer.

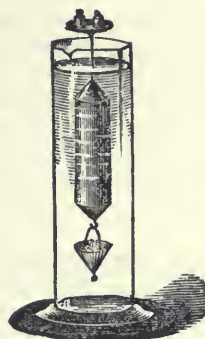


Fig. 28.—Nicholson's hydrometer.

just considered by being arranged to operate at a constant level, but with varying weight. Such a hydrometer has but one mark on tube, and is made to float in all liquids at this point by adding or taking away weights from the pan, as shown in Fig. 28.

On account of the inconvenience of using it, this hydrometer is practically limited to lecture counter demonstration, though at least two modifications of same principle are found very useful forms of specific gravity apparatus.

The first of these is *Rousseau's densimeter*, shown in Fig. 29, in which the scale pan of the Nicholson hydrometer is replaced by a small cup, and into this cup is poured the liquid the specific gravity of which is desired, while the instrument itself is floated in water. From the depth to which the standard amount of liquid sinks the hydrometer, the specific gravity of the liquid can be deduced. The prime value of Rousseau's densimeter is to take the specific gravity of liquids which are either very costly or so rare that a larger quantity, such as is necessary for a pycnometer or a hydrometer, is not obtainable.

The Mohr-Westphal Balance.—In this balance, as in the Nicholson hydrometer, the specific gravity is estimated by the floating of a weighted cylinder which is kept at a constant level by means of applying weights. The balance as shown (Fig. 30) is far more convenient to handle than the Nicholson, inasmuch as the floating cylinder is permanently attached to the end of the scale beam, and a constant level maintained by keeping

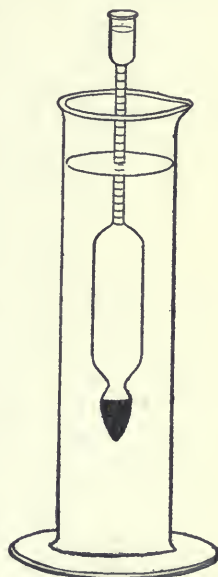


Fig. 29.—Rousseau densimeter.

the balance in equilibrium. In making such a balance a cylinder is made out of small glass tube weighted with an appropriate quantity of mercury, or else a thermometer, and the exact bulk of same is carefully estimated by immersing same in water contained in a burette or other narrow graduated tube, and noting the rise of the water when the cylinder or thermometer is completely immersed.

This cylinder is suspended from one end of the beam, the other end of which is weighted so as to produce equilibrium in air. When the cylinder is immersed in water, the buoyancy of the latter throws the balance out of equilibrium, which, however, can be restored by placing on the beam, directly over the knife-edge from which the cylinder is suspended, an appropriate weight in the form of a rider; this weight must, of course, be exactly that of the weight of the bulk of water, equal to the bulk of the cylinder, for the body immersed in water loses as much weight as the weight of the water displaced.

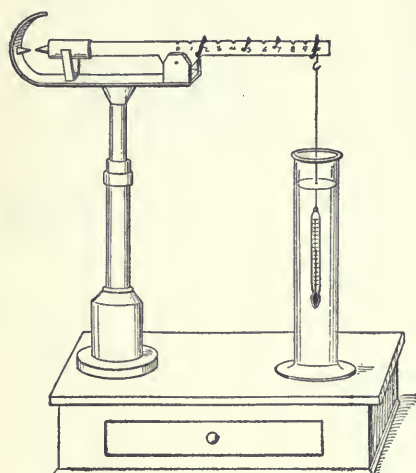


Fig. 30.—Mohr-Westphal balance.

In the Westphal balance the beam is accurately divided into ten equal parts, up and down which the rider can be freely moved. Duplicate of this rider weight is also provided, and likewise two weights, one-tenth and one one-hundredth the value of the original rider weight. By means of these rider weights the specific gravity of any liquid can be accurately determined. Thus, in the picture one large rider is on the notch over the point of suspension, meaning specific gravity 1.000. The second large

rider is on the notch 2 of the beam, corresponding to specific gravity 0.200. The one-tenth weight rider is on notch 5, corresponding to specific gravity 0.05; while the one one-hundredth weight rider is on notch 7, corresponding to specific gravity 0.007. These four weights, therefore, show that the specific gravity of the liquid is 1.257.

Lovi's Beads.—Lovi's beads are one of the simplest forms of hydrometers. They consist of beads of glass so weighted with mercury that a definite proportion exists between the weight of the bead and its volume. Thus, a bead measuring 2 Cc., if weighted with mercury enough to weigh exactly 3 Gm., will be one and one-half times heavier than water, since 2 Cc. of water weighs 2 Gm.

A bead having the capacity of 2 Cc. and weighing $2\frac{1}{2}$ Gm. would be one and one-fourth times heavier than water. A bead having a capacity of 2 Cc. and containing enough mercury to weigh $1\frac{1}{2}$ Gm. would be three-fourths as heavy as water, or have a specific gravity of 0.75, and, lastly, a bead of 2 Cc., containing enough mercury to weigh exactly 2 Gm., would be the same weight as water, or specific gravity 1.000.

After getting their weights accurately adjusted, the beads are sealed and the specific gravities marked on the outside thereof.

If these four beads were thrown into water, the one having specific gravity 0.75, being lighter than the water, would float therein, and the ones of the specific gravities 1.50 and 1.25 would sink, being heavier than the water, while the one of exactly the same specific gravity as the water would float indifferently at any place in the water where immersed. In the same way, if these beads were thrown into a liquid of specific gravity 1.25, the bead marked 1.50 would sink, those of specific gravities 0.75 and 1.00 would float on the surface of the liquid, while the one of specific gravity 1.25 would float indifferently.

Thus are Lovi's beads employed, a handful of them being thrown into the liquid the specific gravity of which is to be ascertained, and the one which floats indifferently in the solution is of necessity of the same specific gravity as the liquid.

The application of such beads is usually limited to the estimation of specific gravity of boiling liquids, in some processes of manufacture it being directed that the liquid be evaporated to a certain specific gravity. If in such a heated liquid a hydrometer is placed, there is more danger of fracture than if Lovi's beads were applied.

Jolly's Spiral Balance.—This balance, described on page 29, can be used instead of the ordinary balance for estimating specific gravity of either solids or liquids. In the case of solids, the weight of same is ascertained in air and then in water, and the well-known rule applied: weight in air divided by loss of weight in water equals specific gravity.

In case of estimating specific gravity of liquids, a sinker whose volume is known is suspended from the balance, and its downward force, its weight, is estimated in air, in water, and in the liquid whose specific gravity is desired to be known. In this way we learn the loss of weight of the sinker in water and in the liquid, and from this data the specific gravity of the liquid in question is deduced.

ESTIMATION OF SPECIFIC GRAVITY OF SOLIDS

The specific gravity of solids can be estimated in several ways: (a) By means of graduated tube; (b) by floating in liquid of known specific gravity; (c) by means of a hydrostatic balance; (d) with a pyknometer.

With Graduated Tube.—Specific gravity is the weight of a substance compared to the weight of an equal bulk of water, and, as cited above, the estimation of the weight of a certain bulk of liquid and comparison of this to the weight of an equal bulk of water means the simple expedient of weighing a certain volume of the liquid and comparing it to the weight of the same volume of water. Taking the specific gravity of a solid is rendered more difficult by reason of the fact that the great majority of masses of solids possess irregular surfaces, making the exact measurement of their cubical capacity an impossible task, while the lack of mobility of their particles, which distinguishes the solid from the liquid, prevents solid masses from assuming the shape of a standard vessel. Hence the weighing of a certain bulk of solid, for comparison with an equal bulk of water, by means of a pyknometer alone, is out of the question. The only method, therefore, of estimating

the volume of the substance as compared to the volume of water is by immersing the solid in the water contained in a graduate tube and noting the rise of the water upon immersion. Thus, if a piece of brass weighing eight grammes immersed in water contained in a burette causes the surface of that water to rise one cubic centimeter, it will mean that one cubic centimeter of this brass weighs eight grammes. We know that one cubic centimeter of water weighs one gramme, and if the brass weighs eight times as much as the water, its specific gravity is, therefore, eight.

By Immersing Solid in Liquid of Unknown Specific Gravity.—We have already learned, under Lovi's beads, that a substance weighing the same as the same bulk of a certain fluid will float indifferently in that fluid, and this principle was used to estimate the specific gravity of a liquid by throwing beads of known specific gravity therein. Inversely, the specific gravity of a solid can be estimated by throwing the solid into liquids of different specific gravities, and noting in which liquid the substance floats, in which it sinks, and, above all, in which it floats indifferently. Thus, if a piece of ore sinks in a solution of mercuric nitrate of specific gravity 2.00 and floats in a solution of potassiomeric iodide of specific gravity 3.00, it can be safely assumed that this ore has a specific gravity somewhere between these two figures, and the exact specific gravity can be ascertained by diluting the last solution with sufficient water until the ore floats indifferently therein, then noting the specific gravity of the liquid by means of the hydrometer.

Such method of estimation is of comparatively limited application, by reason of its lack of convenience, and an accurate estimation by this method takes more time than does the well-known process of weighing in air and then in water. Its chief use, therefore, is limited to mineralogic work, in which case a series of solutions of known specific gravities, ranging from $1\frac{1}{2}$ to 3, are kept constantly on hand, and the estimation of the selected ore made by throwing same into one solution, then into another, until we find the solution in which it floats indifferently.

A pharmaceutical application of this method was suggested by Hager for estimating the specific gravity of wax, he preparing a series of ten strengths of diluted alcohol of specific gravities ranging from 0.950 to 0.970, and the freshly melted drop of wax is thrown into one after the other of these solutions.

The two methods of estimation of specific gravity just given are of little pharmaceutical value, and are cited first merely because they give the principles of density more clearly than the method in general use; that estimation being—

By Means of a Hydrostatic Balance.—In taking the specific gravity of solids by this process four conditions are possible:

First, the solid may be insoluble in, and heavier than, water.

Second, the solid may be insoluble in, and lighter than, water.

Third, the solid may be soluble in, and heavier than, water.

Fourth, the solid may be soluble in, and lighter than, water.

(A) *Solids Insoluble in and Heavier than Water.*—As mentioned above, the direct comparison of the bulk of a mass of solid with the same bulk of water is a matter of some difficulty. Some indirect method had to be devised, and this was reached by a practical application of a theorem of Archimedes, one of the ancient philosophers.

Suppose, into a glass full of water to the brim, an eight-ounce brass weight is dropped; of course, the water will overflow. Now, Archimedes found an interesting comparison between the weight of the overflowing water and the changed weight of the brass in the water. He found that a body in water weighed less than it did in air. The student should demonstrate this for himself, for practical demonstrations are of great value. Take a hand scale and tie a weight beneath the pan; weigh it in the air, then in the water, when it will be found that brass,

which weighs eight ounces in air, weighs only seven ounces in water. It, therefore, loses one ounce of its weight when immersed in water.

Archimedes showed that the amount of water displaced from the full glass mentioned above was one ounce in weight also. Accordingly, he devised his famous theorem, "A body immersed in water loses as much weight as the weight of the water it displaces." Naturally, the amount of water displaced by a body means a bulk equal to that body, and thus we indirectly get the weights of equal bulks of water and the body with which we are experimenting. If the relative weights of equal bulks of water and some other liquid were as 1 is to 8, the specific gravity of the other liquid would be 8 divided by 1, or 8; if the relative weights were, say, 240 grains for water and 480 grains for the other liquid, the specific gravity of the latter would be 480 divided by 240, or 2. Remember that loss of weight of a body in water means the weight of a bulk of water equal to that of the body, and one can easily understand the rule for taking the specific gravity of solids. "Weigh the body in air, then in water. Divide the weight in air by the loss of weight in water, and the quotient will be the specific gravity."

The principle of this case has been considered, but as to practical manipulation, it need only be stated that the substance of which the specific gravity is desired is attached to a balance by a fine silk thread, better, a horsehair, suspended in a beaker, or other suitable glass containing distilled water of definite temperature (usually 15° C.), after being carefully weighed in air.

It will be observed that the body weighs less in water than it did in air, and the difference between these two figures (the loss of weight in water) is, according to the theorem of Archimedes, exactly the same as the weight of the same bulk of water; hence if an 8-gramme brass weight immersed in water weighs 7 grammes, the loss of weight of one gramme can be transcribed into the statement that the quantity of water of exactly the same bulk as the 8-gramme weight would weigh exactly 1 gramme.

If, during this experiment, the brass weight is immersed in a graduated tube, it will be found that, on placing the 8-gramme brass weight below the surface of the water, the water in the tube will rise exactly one cubic centimeter if the temperature of the water is 4° C. Now, one cubic centimeter of water weighs one gramme, and, therefore, we learn that the loss in weight in water of a given substance is exactly equal to the weight of an equal bulk of water.

This being the case, we see that, by estimating the loss of weight in water, we have a direct comparison of the weight of the substance with the weight of an equal bulk of water, and all that is necessary in order to deduce the specific gravity is to divide the weight in air by the loss of weight in water, that is, divide the weight in air by the weight of an equal bulk of water, it being always remembered that the specific gravity means how many times heavier is a substance than is the same bulk of water. Hence, memorize this rule: *Weight in air, divided by loss of weight in water, equals specific gravity.*

(B) *Solids Insoluble and Lighter than Water.*—Such solid of necessity floats on the surface of the water, and in order to find the loss of weight in water the substance must be completely immersed. In estimating the specific gravity of such solids they must be forced under the surface

of the water by attaching some heavy substance, for example, a brass weight.

The method of taking the specific gravity of the substance after immersion is practically the same as that of taking the specific gravity of a solid insoluble in and heavier than water, namely: we must know the weight of the substance and the sinker separately in air, the weight of both in water, and the weight of the sinker alone in water.

The weight of the two substances, minus the weight of both in water, gives the loss of both in water, and from this figure the loss of the weight of the sinker alone is taken; we then have the loss of weight of the lighter substance in water, whereupon we apply the rule: *Weight in air divided by loss of weight in water equals specific gravity.*

An illustration of such an estimation is as follows: A piece of wax weighs 4 grammes in air, and to immerse same, we attach a brass weight weighing 50 grammes. The two weigh in water 43.84 grammes; hence the loss of the weight of the two in water equals $54 - 43.84$, or 10.16 grammes.

Then we immerse the sinker in water and find it weighs 44 grammes. Then the weight of the sinker in air (50 grammes), minus the weight of the sinker in water (44 grammes), equals the loss of weight of the sinker in water (6 grammes). The combined loss of weight (10.16 grammes) minus the loss of the sinker alone (6 grammes) gives the loss of the weight of wax in water (4.16 grammes). The weight of the wax in air (4 grammes), divided by the loss of the weight in water (4.16 grammes), gives the specific gravity (0.960).

In observing these figures, the student is confronted with what, at a first glance, seems a peculiar statement, that a piece of wax weighing 4 grammes loses, when immersed in water, 4.16 grammes; that is, it loses more weight than it actually possessed. This can be easily explained, however, when we appreciate the fact that weight is merely the measure of the gravitating force of the earth, and that when a substance hanging toward the earth is immersed in a liquid, it is acted upon by an opposing upward force, namely, the buoyant force of water. This explains why, when a substance is immersed in water, it is said to lose weight, the so-called loss of weight being the difference between the actual attractive force of the earth and that same force when modified by the upward force of the liquid in which the substance is immersed.

In case of solids lighter than water the upward buoyant force of the liquid is greater than the downward force of gravitation, hence the phenomenon of a body losing more weight than it originally possessed.

(C) *Solids Soluble in and Heavier than Water.*—It is hardly necessary to state that with some substances (lump-sugar, for example) we cannot accurately estimate the loss of the weight in water, for while we are endeavoring to weigh the substance in water, the water will be dissolving the solid, which will finally disappear under the solvent action of the liquid; in such cases, therefore, we must have recourse to an indirect estimation, namely: by estimating the loss of weight of the substance in some liquid in which it is insoluble, and, from this, estimating what would be the loss of weight of the same substance in water by a proportion involving the respective specific gravities of the two liquids.

Thus, if a piece of alum weighs in air 3 grammes, and weighs in turpentine (sp. gr. 0.87) 1.48 grammes, the loss of weight in turpentine will be 1.52 grammes. If we divide the weight in air by the loss of weight in turpentine, we would estimate how many times heavier the substance was than turpentine, which datum is of no interest to the observer. In other words, the loss of weight in turpentine represents the weight of an equal bulk of turpentine, while what we really want to know is the weight of an equal bulk of water (or loss of weight in water).

This is estimated by the following proportion:

The specific gravity of turpentine is to specific gravity of water as the loss of weight in turpentine is to the loss of weight in water, and applying this to the above example, we have:

$$\begin{aligned} 0.87:1.00::1.52:\times \\ \times = \frac{1.00 \times 1.52}{0.87} = 1.74. \end{aligned}$$

By this we see that the piece of alum, which lost 1.52 grammes in turpentine, would lose 1.74 grammes if it were possible to estimate the loss of weight in water by immersing same therein. This obtained, we apply the usual rule:

Weight in air divided by loss of weight in water equals specific gravity, and get— $3.00 \div 1.74 = 1.73$. (Specific gravity of the alum.)

(D) *Solids Soluble in and Lighter than Water*.—Of this comparatively rare class of bodies it need only be said that the estimation of the specific gravity of same is carried on by combining the two previous methods of estimation, viz., to the substance we attach a weight or sinker and immerse same in some liquid in which it is insoluble; in this way we have a double estimation to perform: First, to subtract the loss of weight of the sinker from the combined loss of weight, as in B. Take the loss of weight of the substance alone in the liquid, and deduce therefrom the loss of weight in water, as in C. Lastly, apply the rule: *Weight in air divided by loss of weight in water equals specific gravity*.

With the Pycnometer.—The estimation of specific gravity of solids by means of the balance is limited to such substances possessing sufficient cohesion to enable same to be suspended from a thread or horsehair, a method which is inapplicable to powders.

In estimating the specific gravity of powders, therefore, recourse is had to a modification of the pycnometer experiment, which can be best explained by citing a possible case.

Assuming we had powdered calomel weighing 10 grammes, we would take the specific gravity of same in a pycnometer, holding, say, 100 grammes distilled water. Now, if all the 100 grammes of water would remain in the pycnometer on the addition of calomel, the combined weight would be, of course, 110 grammes. However, it is an accepted fact that when the powdered calomel is placed in a bottle, water will be displaced and some rejected, the amount of rejected water being equal to the weight of an equal bulk of calomel. In the actual experiment the weight of the 10 grammes of calomel and of water sufficient to fill the 100-gramme bottle was found to be 108.6 grammes. This shows that the amount of water displaced is $110 - 108.6$, or 1.4 grammes. In other words, the weight of a bulk of water equal to the bulk of the 10 grammes calomel was 1.4 grammes; hence the calomel is as many times as heavy as water as 1.4 goes into 10; hence the specific gravity of the calomel is $10 \div 1.4$, or 7.1.

As mentioned on page 47, the knowledge of the specific gravity of pharmaceutic substances aids us in estimating the volume of a certain weight of liquid.

Thus, if we know that the specific gravity of sulphuric acid is 1.83, and we have a recipe calling for a given weight of same, we can transcribe this weight into the liquid volume of the acid by dividing the number of grammes directed by the specific gravity of the acid. In the same way if we wish to know what size container is necessary to hold twenty avoirdupois pounds of glycerin (sp. gr. 1.25), we can find out by a simple calculation involving the number of gallons in twenty pounds of water and dividing this number by the specific gravity of glycerin. This latter example, however, is perhaps more properly solved by specific volume, mention of which will be made shortly.

In answer to the question as to the pharmaceuticals of which we should study the specific gravities, it might be stated that every pharmacist should know at least the following:

Ether.....	0.716	These figures represent the specific gravity of each substance at 25° C. (77° F.), for bear in mind that temperature is an important factor in deducing specific gravity—the warmer the temperature, the less dense the liquid.
Chloroform.....	1.476	
Glycerin.....	1.246	
Alcohol.....	0.809	
Sulphuric acid.....	1.826	
Nitric acid.....	1.403	
Mercury.....	13.535	

A practical demonstration of the use of specific gravity may be valuable. We want to know how many grains there are in a fluidounce of ether, specific gravity 0.720.

One fluidounce of distilled water weighs 455.7 grains; therefore a fluidounce of ether, sp. gr. 0.720, weighs $455.7 \times 0.720 = 328.1$ grains.

$$\begin{array}{r}
 455.7 \\
 \times .72 \\
 \hline
 9114 \\
 31899 \\
 \hline
 328.104
 \end{array}$$

SPECIFIC VOLUME

Specific volume is just the reverse of specific gravity.

Specific gravity is the relative weight of equal bulks of different bodies.

Specific volume is the relative bulk of equal weights of different bodies.

To get the specific volume of a body we divide 1.000 by the specific gravity. Thus, the specific volume of chloroform is approximately 0.666 +

$$1.000 \div 1.50 = 0.666 +.$$

As a homely example, while specific gravity means, in a general way, how many avoirdupois ounces, grammes, etc., in one pint, liter, etc., of alcohol, chloroform, etc., specific volume teaches what size bottle it will take to hold a pound or a kilogramme of the substance in question. Thus specific gravity deals with what weight of a certain substance will go into a certain sized bottle, while specific volume asks what sized bottle will hold a certain weight of the substance. If we say chloroform has the specific gravity 1.50, we mean (since pint and avoirdupois pound of water are somewhat approximate) that a pint

of chloroform weighs somewhere around $1\frac{1}{2}$ avoirdupois pounds. Regarding this same chloroform, an avoirdupois pound of it will not, as is full well known, fill a pint bottle. In fact, it will easily go into a twelve-fluidounce bottle. We get nearer its exact bulk by dividing 16 by the specific gravity, or, in the present case, 16 divided by 1.50 equals 10.6, and we find that one avoirdupois pound of chloroform has a bulk of about 10.6 fluidounces. This is only approximate, for, in truth, a pint of water weighs more than an avoirdupois pound, and, again, the specific gravity of chloroform is 1.49 (at 15° C.), not 1.50. The exact estimation of the weight of a pint and the volume of an avoirdupois pound of chloroform is as follows:

1 pint	= 16 fluidounces.
1 fluidounce distilled water	= 455.7 grains.
1 pint (16 fluidounces) distilled water	= 455.7 grains $\times$ 16 = 7291.2 grains.
16 fluidounces chloroform (sp. gr. 1.49)	= 7291.2 $\times$ 1.49 = 10,863.888 grains.
7000 grains	= 1 avoirdupois pound.

Therefore, in 10,863.888 grains there are as many avoirdupois pounds as 7000 goes into 10,863.888:

$$10,863.888 \text{ divided by } 7000 = 1.552,$$

and we see that one pint of chloroform weighs 1.552 avoirdupois pounds, considerably over $1\frac{1}{2}$ avoirdupois pounds.

Now for the volume of one avoirdupois pound of chloroform:

1.552 avoirdupois pounds of chloroform measure 16 fluidounces.

1 avoirdupois pound of chloroform will measure $\frac{16}{1.552} \times 16 = 10.3$ ounces—

$$\begin{array}{r} 1552 \overline{)16000(10.3 +} \\ \underline{1552} \\ 4800 \\ \underline{4656} \\ 144 \end{array}$$

Before closing this chapter, other phases of simple arithmetic as applied in pharmacy may well be taken up.

ALLIGATION

Alligation, or "the rule of mixtures" as applied in pharmacy, includes those arithmetic processes used in ascertaining the relative amount of different strengths of the same substance needed to blend into a substance of a definite intermediate strength. Thus, by alligation we solve such problems as the blending of two strengths of opiums into a third strength.

Opium, as we will learn later, is a substance whose activity is based on the amount of morphine it contains, and when we say 10 per cent. opium, we mean that 100 grammes of the drug contain 10 grammes morphine; by 18 per cent. opium we mean one that contains 18 grammes morphine to each 100 grammes; while 14 per cent. opium is such as contains 14 grammes to the hundred. It can be seen that we can combine 10 per cent. opium and 18 per cent. opium to make 14 per cent. opium, nor is it difficult to figure out that equal quantities of 10 per cent. and 18 per cent. will make 14 per cent.; but upon what principle is the arithmetic calculation based?

The best comparison of alligation is with the principle of the balance, as fully explained on p. 36. What do we mean by a single equal arm balance? It means a beam represented by the line $a-b$ (Fig. 31) of the same weight throughout, suspended on a central knife-edge, with pans hanging from the end knife-edges (a and b respectively).

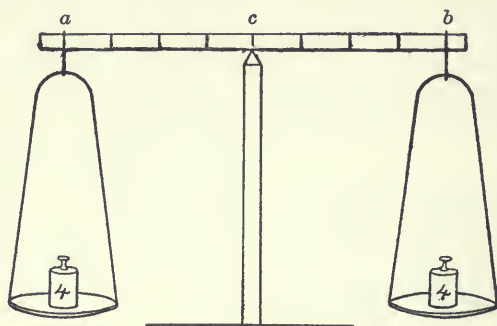
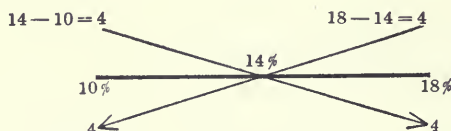


Fig. 31.—Alligation I.

In order that the balance swing exactly even it is necessary that the central knife-edge, c , be exactly midway between a and b . Thus, if we call a 10 inches, and b 18 inches, the beam would be $18-10$, or 8 inches long, and if c be at 14 inches, it will stand $18-14$, or 4 inches, from b , and $14-10$, or 4 inches, from a , and 4 pounds placed on pan a would be exactly balanced by 4 pounds placed on pan b .

Compare this to the following opium alligation. We have 10 and 18 per cent. opiums and wish to mix them so as to make 14 per cent. This we solve as follows:



And we see that 4 pounds of 10 per cent. and 4 pounds of 18 per cent. give 8 pounds of 14 per cent.

Again, what do we mean by a single beam unequal arm balance? we mean a beam, like Fig. 31, only with knife-edge c *not* midway between a and b (Fig. 32).

Assuming as before that we call a 10 inches and b 18 inches, we would have a beam 8 inches long, and if c were at 12 inches, it would stand $18-12$, or 6 inches, from b , and $12-10$, or 2 inches, from a . In order to balance such a beam different weights must be placed on pans a and b , following the general principles of the lever; namely, a certain weight placed on the pan of the long arm b will exert an increased power on the weight in the pan of the short arm a , and in direct proportion to the length of the arms. Thus, in Fig. 32 the long arm $b-c$ (6 inches) is 3 times longer than the short arm $a-c$ (2

inches); hence 1 pound placed on pan *b* will balance 3 pounds placed on pan *a*, or 2 pounds in pan *b* will balance 6 pounds in pan *a*. Compare this to the following opium alligation. We have 10 and 18 per

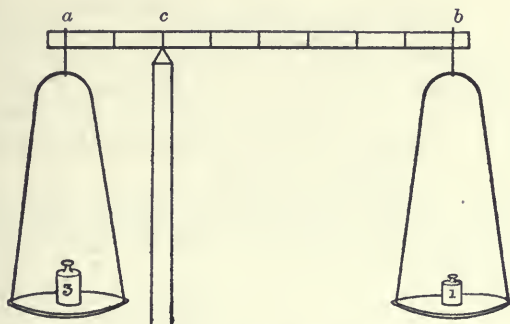


Fig. 32.—Alligation II.

cent. opiums and desire to mix them so as to make 12 per cent. This is solved as follows:

$$\begin{array}{ccc}
 12 - 10 = 2 & & 18 - 12 = 6 \\
 & \searrow & \nearrow \\
 & 12\% & \\
 & \nearrow & \searrow \\
 10\% & & 18\% \\
 6 \leftarrow & & \rightarrow 2
 \end{array}$$

And we see that 6 pounds of 10 percent. and 2 pounds of 18 per cent. give 8 pounds of 12 per cent.

Likewise, assuming our beam has different dimensions, as follows: A, 9 inches; B, 14 inches; C, 12 inches. The beam will then be 14-9, or 5 inches long, and *c* being 12-9, or 3 inches from *a*, and 14-12,

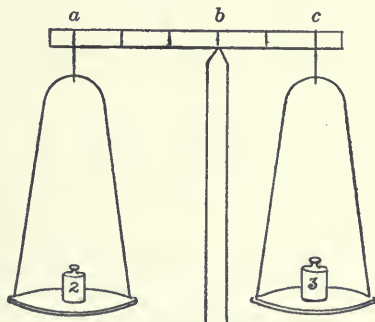
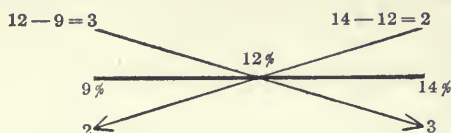


Fig. 33.—Alligation III.

or 2 inches, from *b*, then the weight of 3 pounds placed on pan *b* (14 inches) will be balanced by 2 pounds placed on pan *a* (9 inches).

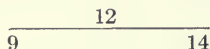
Compare this to the following alligation:

We have 9 and 14 per cent. opiums and want to make 12 per cent. This we solve below:



And we see that 2 pounds 9 per cent. and 3 pounds 14 per cent. give 5 pounds 12 per cent.

Thus, can every problem in alligation be solved, and when we draw our line, with the percentage wanted, above, and the percentages we have, below, like this—



we are unconsciously comparing the matter to a single beam balance. A purely arithmetic solution of alligation is the following tiresome but lucid process.

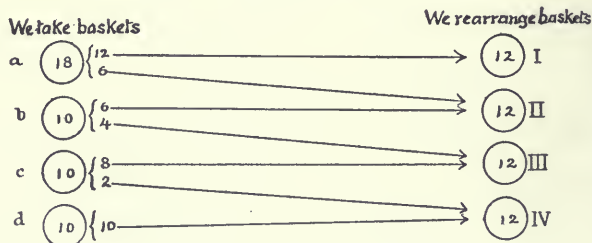
Suppose we take the problem given above: How much 10 and 18 per cent. opiums are needed to make 12 per cent.?

10 per cent. opium means 10 parts morphine to 100 parts of opium.
 18 " " " 18 " " 100 " "
 12 " " " 12 " " 100 " "

These figures are exactly analogous to:

10 apples in a basket,
 18 " "
 12 " "

and as if the question were, How many baskets of 10 apples each and 18 apples each must be taken to arrange baskets containing 12 apples each? Let us do the actual rearranging:



One basket of 18 apples. This gives us one basket of 12 apples (No. I) and 6 apples over. These 6 and 6 from a basket of 10 apples, which we bring here, will make another basket of 12 apples (No. II). Six apples from 10 leaves 4 apples in our basket b and, bringing up basket c (10 apples) we take out 8, making basket No. III contain 12 apples. This leaves 2 apples in basket c, which, plus 10 apples from basket d makes 12 apples to put into basket IV, and the trick is done.

The conclusion is that 1 basket of 18 apples and 3 baskets of 10 apples each give 4 baskets of 12 apples each; the same result as the similar alligation, which showed that—

6 pounds of 10 per cent. (or 3 pounds of 10 per cent.).
 and 2 pounds of 18 " (or 1 pound of 18 per cent.).
 give 8 pounds of 12 " (or 4 pounds of 12 per cent.).

For the benefit of those knowing algebra the following algebraic solution of these kind of problems is given:

Let x represent quantity of the lowest percentage used.
 Let y " " " highest percentage used.
 Let z " " " percentage desired.

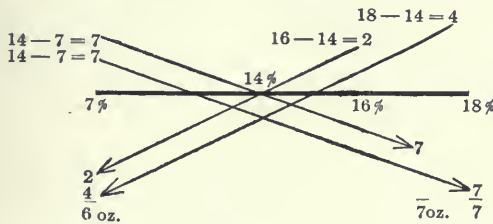
Then the problem, How much 10 and 18 per cent. opiums are needed to make 12 per cent.? is solved as follows:

$$\begin{aligned} 10x + 18y &= 12z. & z &= x + y. \\ 10x + 18y &= 12x + 12y. \\ 18y - 12y &= 12x - 10x. \\ 6y &= 2x. & x &= 3y. \end{aligned}$$

Then, if $x + y = 4$ pounds, and $x = 3y$, $3y + y = 4$ pounds, or $4y = 4$ pounds.

$x = 3y$. $y = 1$ pound. $x = 3$ pounds.
 Hence, 3 pounds 10 per cent.
 and 1 pound 18 per cent.
 give 4 pounds 12 per cent.

If the problem involves three different percentages, say, 7 per cent., 16 per cent., and 18 per cent., out of which to make another percentage, say, 14 per cent., the calculation is accomplished by means of the mechanic "rule of the balance" explained above, viz.,



That is: 6 ounces of 7 per cent. opium.
 7 " " 16 " "
 7 " " 18 " "
 make 20 ounces of 14 per cent. opium.

And if the problem was, to make 14 per cent. opium from 5 per cent., 15 per cent., 16 per cent., and 18 per cent. opium, the calculation would be as follows:

14 - 5 = 9. This goes under 15 per cent., 16 per cent., and 18 per cent.
 15 - 14 = 1. This goes under 5 per cent.
 16 - 14 = 2. This goes under 5 per cent.
 18 - 14 = 4. This goes under 5 per cent.

5 per cent.	15 per cent.	16 per cent.	18 per cent.
1	9	9	9
2			
4			
7	9	9	9

That is: 7 ounces of 5 per cent. opium.
 9 " 15 " "
 9 " 16 " "
 9 " 18 " "
 make 34 ounces of 14 per cent.

In explanation of the fact that in the examples furnished above we find two, and in the other case three, separate quantities of lower percentage which must be added to the stronger, let it be said that the example can be considered as three separate examples or problems, viz.:

(A) How much 5 per cent. and 15 per cent. needed to make 14 per cent.?

Answer by simple calculation: 1 ounce of 5 per cent. and 9 ounces of 15 per cent.

(B) How much 5 per cent. and 16 per cent. needed to make 14 per cent.?

Answered by simple calculation: 2 ounces of 5 per cent. and 9 ounces of 16 per cent.

(C) How much 5 per cent. and 18 per cent. needed to make 14 per cent.?

Answered by simple calculation: 4 ounces of 5 per cent. and 9 ounces of 18 per cent.

We, therefore, have three separate batches of 14 per cent. opium, all made from 5 per cent., but with a higher percentage differing in every case. If we took these three separate mixings and thoroughly blended same, we would, of necessity, have 14 per cent. opium, and this 14 per cent. opium would originally have been made up of—1 ounce 5 per cent. + 9 ounces 15 per cent. + 2 ounces 5 per cent. + 9 ounces 16 per cent. + 4 ounces 5 per cent. + 9 ounces 18 per cent.; in all, 7 ounces 5 per cent., 9 ounces 15 per cent., 9 ounces 16 per cent., and 9 ounces 18 per cent., which we notice is the answer to the example as originally given.

The same method of calculation for alligation can be applied to specific gravity examples, such as: How much glycerin, specific gravity 1.25, and alcohol, specific gravity, 0.820, must be mixed to make a liquid of specific gravity 1.000? which is solved as follows:

$$\begin{array}{rcl}
 & 1.00 & \\
 \hline
 1.25 & & 0.82 \quad \text{or, preferably,}
 \end{array}$$

$$\begin{array}{rcl}
 125 - 100 = 25 & & 100 - 82 = 18 \\
 \swarrow & & \searrow \\
 125 & 100 & 82 \\
 \nwarrow & & \swarrow \\
 18 & & 25
 \end{array}$$

Answer: 18 ounces glycerin
 and 25 ounces alcohol
 make 43 ounces of liquid, sp. gr. 100.

This example and also the percentage examples can be proved by the following reasonings:

18 fluidounces glycerin sp. gr. 1.25 = $18 \times 455.7 \times 1.25 = 10253.2$ grains.
 25 fluidounces alcohol sp. gr. 0.82 = $25 \times 455.7 \times 0.82 = 9341.9$ grains.
 43 fluidounces of liquid weigh 19595.1 grains.
 43 fluidounces of water weigh $43 \times 455.7 = 19595.1$ grains.

Hence the mixture has same specific gravity as water, since 43 fluidounces of it weigh the same as 43 fluidounces of water.

The same method is used to solve such examples as:

What will be the specific gravity of a mixture of 5 fluidounces of solution of tersulphate of iron, specific gravity 1.555, and 3 fluidounces water? The figuring being as follows:

5 fluidounces of sol. tersulph. iron = $5 \times 455.7 \times 1.555 = 3543.0$ grains.
 3 fluidounces of water = $3 \times 455.7 \times 1.000 = 1367.1$ grains.
 8 fluidounces of liquid weigh 4910.1 grains.
 8 fluidounces of water weigh $8 \times 455.7 = 3645.6$

Hence the liquid weighs $\frac{4910.1}{3645.6}$ as much as water and has the specific gravity 1.346.

While speaking of such examples, reference should be made to the simple example involving percentage solutions, such as: How much cocaine is needed for one fluidounce of a 5 per cent. solution?

The absolutely accurate way of solving this is as follows:

Weight fluidounces water }
 455.7 grains } $\times 0.05 = 22.785$ grains,

which represents the number of grains cocaine (hydrochloride presumably) needed to make the solution.

In this case, however, in order to obtain accurate results, the 22.785 grains cocaine hydrochloride, must be weighed out, dissolved in water, and finished by bringing the finished solution up to 455.7 grains by weight, which, of course, will measure slightly less than one fluidounce.

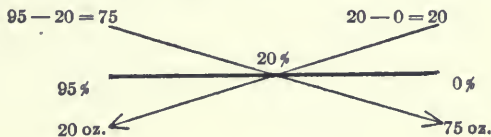
Another and less accurate method, very largely employed, is by taking 5 per cent. of 480 minims, or 24 grains, dissolving this in water to make a fluidounce. This, while by no means as accurate as the former process, is more convenient, and can be safely employed in preparing solutions in small percentages.

A simple modification of alligation is that of percentages outlined in the last pharmacopœia under alcohol, where a rule of dilution is given. The rule is practically as follows:

If we have a 95 per cent. alcohol and want to dilute it to 20 per cent., we must then take 20 fluidounces of the 95 per cent. and add enough water to make 95 fluidounces; when we will have approximately 20 per cent. alcohol.

It may also be solved by the regular process of alligation if we recast the problem to the following wording: How much 95 per cent. alcohol and 0° alcohol (water) needed to make 20 per cent. alcohol?

Solved by "rule of the balance."



This shows that we can reduce 95 per cent. alcohol to 20 per cent. by reversing the fluidounces with the percentages. That is, from 20 fluidounces of 95 per cent. alcohol we can make 95 fluidounces of 20 per cent. But suppose the question is: How much 20 per cent. alcohol could be made from 5 fluidounces 95 per cent.? It is a case of simple proportion.

If 20 fluidounces 95 per cent. give 95 fluidounces 20 per cent., 5 fluidounces 95 per cent. ($\frac{1}{4}$ of 20) will give us $\frac{1}{4}$ of 95 fluidounces 20 per cent., or $23\frac{3}{4}$ fluidounces.

Expressed in proportion:

$$\begin{array}{l} 20 : 5 :: 95 : \times \\ \times = \frac{5 \times 95}{20} = 23\frac{3}{4} \text{ fluidounces.} \end{array}$$

It is important that this be understood. The rule, by the way, applies *exactly* only when there is no diminution in volume.

CHAPTER IV

HEAT

HEAT is that form of molecular motion which produces the physiologic sensation we call warmth, this molecular motion being produced by friction, electricity, light, or chemical action. In order to obtain a clear understanding of heat, a comprehension of the molecule as explained on p. 359 is essential. Suffice it to say here that molecules are very minute particles of matter, and these molecules, working among themselves, produce friction. That *friction* is the source of heat is well known, and can be demonstrated by such simple experiments as rubbing one surface on another when both become hot. This is shown by rubbing the finger rapidly on a piece of wood, when the sensation of warmth is transmitted to the body. It is also known that when any metallic body, such as an axle, rubs rapidly on its bearings, if the axle is not well greased to prevent this friction, the heat produced is sufficient to set fire to the surrounding objects, producing the condition called "hot box." Primitive people, such as Indians, becoming adept at the art, were able to kindle fires by rubbing two pieces of wood together.

In each case we have a striking illustration of friction as a source of heat.

Another source of heat is *electricity*, and in such cases the friction of the molecules of the non-conductor produces heat. In many cases the resistance causes the substance to glow or become incandescent. Such is the basis of the incandescent electric light in which the electricity is forced through a filament of poorly conducting carbon, the molecules of carbon, in resisting the electric pressure, being heated sufficiently to become incandescent.

A discussion of the principles of electricity is beyond the scope of this work, and for details of this force the reader is referred to any standard text-book of physics.

Suffice it here to state that electricity is a molecular condition, and that this condition is transmitted from one molecule to another with more or less rapidity, dependent on the character of the substance influenced. Matter which rapidly transmits the electric condition from one molecule to another is called a conductor of electricity, while matter the molecules of which do not transmit the condition is said to be a non-conductor or a poor conductor.

As the question of conduction of electricity is a purely relative matter,—all bodies transmitting the condition to an extent more or less great,—the terms above mentioned are gradually becoming obsolete, and the words “conductivity” and “resistance” being substituted to cover the respective meanings. Remember that electricity is a molecular condition, and not an entity, and, therefore, when we talk of “the electric current” passing through a wire, we mean that the electric condition affecting the end molecule of the wire is rapidly transmitted to the adjoining molecule, and thence from one molecule to another, the whole length of the wire. This being clearly understood, we can use the simpler term, “passage of the electric current,” in the following explanations.

Electricity passes rapidly from molecule to molecule through a conductor; but if compelled to run through a non-conducting substance, it requires a current of high potential, of great force, to overcome the resistance of this body.

Light also affords a minute amount of heat. Heat, light, and electricity are so-called physical forces affecting molecules, and are, under certain circumstances, convertible one into another.

We have just seen that electricity is converted into heat; inversely, heat can be converted into electricity.

By applying heat to two metal plates in close contact with each other, an electric current is formed in the apparatus known as the “pyroelectric battery.” Hence it is not strange that light can be converted into heat, although the amount produced is very minute and very difficult to measure, inasmuch as almost invariably all sources of light are also sources of heat, and, therefore, it is very difficult to isolate light from its accompanying heat.

Chemical action is the chief source of heat. This phenomenon, which is fully described on p. 360, means a breaking up of the molecules of a substance, with the formation of new molecules. This decomposition is accompanied with considerable movement of the molecules and their constituent atoms, thus producing a form of friction and, therefore, producing heat.

Combustion.—Most chemicals reacting on one another produce heat, although in some cases the reverse is true. As an illustration of heat produced by chemical action, iron combined with iodine in a test-tube in the presence of water will so heat the test-tube as to render it difficult to hold same in the hand. Likewise, sulphuric acid combined with solution of soda produces great heat, and numberless other illustrations could be given. The production of heat by such chemical action is different from the chemical action in the burning of wood.

The invariable accompaniment of the latter being flame, such form of chemical action is called *combustion*; that is, chemical action accompanied by heat and *light*. We know that wood is composed of hydrogen, oxygen, and carbon; that the air around us contains oxygen, and that by applying a flame to the wood there is commenced a chemical reaction which results in the formation, from the carbon hydrogen and oxygen that is present, of carbon dioxide (CO_2) and of water (H_2O).

In this chemical reaction, as in the formation of ferrous iodide, heat is produced, and of much greater intensity. So great is the heat that some of the carbon, which in the hurry of the reaction escapes combination with the oxygen, glows with heat, just as does the carbon filament in the incandescent light. To prove the presence of incandescent carbon hold a plate over any luminous flame, when it will become blackened with soot, which is pure carbon. Whenever the combustion—as this reaction, in which the carbon and hydrogen of a substance combine with the oxygen of the air, is called—is accompanied by unchanged carbon glowing with heat, a flame is produced.

Any substance with which we can perform a combustion is called fuel. Such matter is very abundant, and is found in all three states of aggregation—solids, liquids, and gases.

Solid Fuels.—The solid fuels are wood, coal, and charcoal. Wood contains hydrogen and oxygen besides carbon, whereas coal and charcoal consist very largely of carbon. In the case of each fuel the chemical action involved is the combination of the carbon with the oxygen of the air, forming carbon monoxide and carbon dioxide, with more or less particles of unconsumed carbon, called soot.

In burning solid fuels the apparatus designed to contain same is called stove or furnace, and consists fundamentally of the fire-box in which the fuel is held, the chimney or flues for removal of the gaseous products of combustion, and damper or vents for the admission of air necessary to produce the action. Provision is made, by means of grating, for the removal and storage of ashes, which constitute the inorganic salts present in wood or coal.

In order to produce successful combustion the oxygen of the air is absolutely necessary, hence if a furnace or stove is not supplied with sufficient air, the fire within the box will be “smothered” or “go out.” This explains the method of smothering a fire by surrounding the burning substance with blankets, thus preventing access of air. In order to produce good combustion, a comparatively good draft of air must be had, the intensity of the draft being regulated by the opening or closing of the vents or dampers, and in large furnaces by the relative height of the chimney or smoke-stack.

For the practical application of combustion of solid fuels the reader is referred to any kitchen-range or house-furnace.

The **liquids** used as fuels are alcohol and petroleum products (gasoline and insurance oil, etc.). In using liquids as fuel there are two methods employed:

If the liquid is not very volatile, such as insurance oil, astral oil, etc., the liquid is sucked up through a wick by capillary attraction, and burned as a liquid off the top of the wick. Such a method is also employed in the burning of the volatile liquid alcohol in the ordinary alcohol lamp (Fig. 34).

In cases where the liquid is quite volatile (as gasoline), the apparatus used for the purpose is so arranged that the liquid is first converted into vapor and this vapor burned. Such is the principle of the vapor stove (Fig. 35), which possesses a great advantage over the wick stoves by having the vapor burned mixed with air, as in the Bunsen burner, thus preventing a sooty flame. Alcohol is also burned in the form of vapor in the so-called "Russian blast lamp," which produces an intensely hot flame (Fig. 36).

Gaseous Fuels.—The gases used to produce heat are coal-gas, acetylene, and natural gas, the first two being manufactured, while the latter is obtained in certain sections of the world by simply sinking



Fig. 34.—Spirit-lamp.

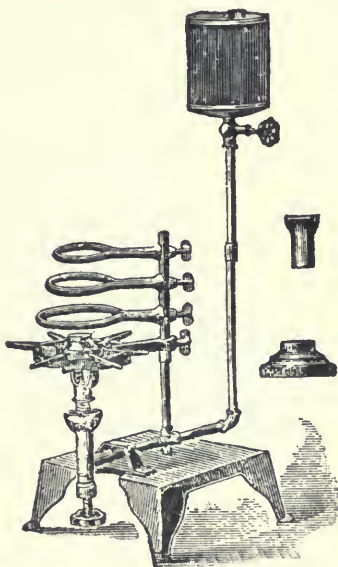


Fig. 35.—Vapor stove and burner.

iron tubes, through which the gas escapes from pockets under the earth's surface.

As to the manufacture of artificial gas, coal-gas is made by the destructive distillation of coal; details of the manufacture can be found in any advanced work on technical chemistry. Acetylene is produced by the action of water on calcium carbide.

Chemically, these fuel gases resemble one another, all being combinations of carbon, which furnishes the heat and light when consumed in the presence of air in the operation of combustion in different forms of burners, according as the intention is to produce a luminous or a non-luminous flame, the latter being used for heating purposes.

A luminous flame is produced in the so-called "fish-tail" burner, in which the gas, as it comes in contact with the air, is ignited by a match or other flame, burning to carbon dioxide and water with incomplete combustion; that is, in the reaction all the carbon is not consumed, and the unconsumed carbon, through the influence of the

intense heat generated by the chemical reaction, glows with heat, or, technically expressed, becomes "incandescent" (Fig. 37).

Why can some part of the carbon escape chemical combustion?

Bear in mind that the combination is with the oxygen of the air; that the gas evolving from a fish-tail burner forms a mass of some thickness. The oxygen of the air can combine only with the outer surface of this mass, leaving the inside of the flame untouched; just as a tightly closed book, when immersed in water, will be soaking wet on the outside and comparatively dry inside. That gas inside the flame has not enough oxygen to convert all the carbon into carbonic oxide, and the result is that much of the carbon remains unchanged, glowing with the heat of the ardent reaction, and depositing as soot when it passes from the flame. The fish-tail flame (Fig. 37) may be quaintly likened to a chemical factory. Zone *a* is the reception room, when the crude material that is to be worked up—the gas from the mains—comes in; zone *b* is the workshop where the chemical reaction is carried on with the utmost vigor—where the hydrocarbon of which gas is composed is broken into carbon and hydrogen, and these in turn combine with the oxygen of the air to form carbon dioxide and water; while



Fig. 36.—Russian alcohol blast lamp.

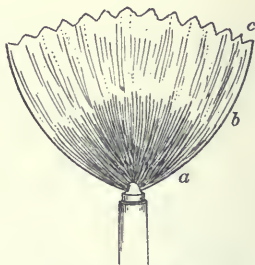


Fig. 37.—Fish-tail flame.

zone *c* is the shipping-room, whence the finished products just mentioned are sent away. Since the soot is dirty and inconvenient in cooking, efforts were made to so construct a burner that the gas would have contact with the air (or its oxygen, for air is composed of nitrogen, oxygen, and some carbon dioxide), in parts other than the outer surface, and this is done by mixing the gas with air before burning it in the Bunsen burner. Aside from the great advantage of freedom from soot, the flame of the Bunsen burner is superior to the ordinary flame by reason of its greater heat. In it all the carbon of the gas becomes combined with the oxygen of the air, and it is self-evident the more chemical reaction occurs in a certain space (as in the mass of gas issuing from a burner), the greater will be the heat produced.

With this purpose in view the Bunsen burner is constructed with a few modifications, according to the purpose adapted. In its simplest form it consists of an upright metallic tube with orifices at the bottom, and as the gas rushes upward through the tube, it mixes with air that enters through the bottom orifices, and when emerging from the top of the tube and ignited, it contains sufficient air thoroughly to combine with all the carbon, thus producing carbon dioxide and water, and no

unconsumed carbon or soot. A practical form of this type is seen in Fig. 38.

In using such a Bunsen burner the flame sometimes "strikes back," that is, the gas which is ignited at the top of the tube finally catches fire at the bottom of the tube, where the unmixed gas emerges, thus producing a sooty flame, and at the same time heating the brass tube, sometimes to a dangerous extent. Such a striking back can be avoided only by having a good current of gas turned on. It can usually be detected from a distance by the peculiar odor of acetylene generated in this incomplete combustion.

For the purpose of more extended heating, larger varieties of the so-called gas-stove are obtainable, the most practical of which, from the pharmacist's standpoint, being the Fletcher radial burner shown in Fig. 39, in which the gas is mixed with the air in a horizontal tube, and emerges through radiating slits cut into a central dome, thus making a flat, clean, and intensely hot flame.

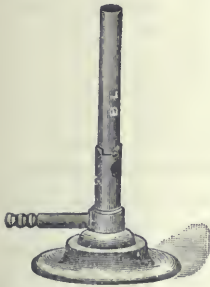


Fig. 38.—Bunsen burner.

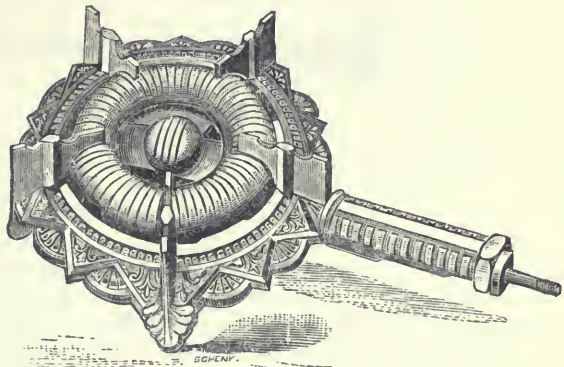


Fig. 39.—Fletcher's radial burner.

In the old-fashioned form of gas-stove the flame used was a luminous one, and the blackening of the utensils through soot was avoided by placing between the utensil and the flame a wire gauze.

The advantage a Bunsen burner possesses over the ordinary fish-tail burner is due to the fact that the gas before ignition is mixed with sufficient air to produce a complete combustion, and this is of benefit—first, in producing a smokeless flame, and, second, in giving a more intense heat than is produced by the same gas when burned in a fish-tail burner.

That more heat is produced is self-evident when we consider that the heat generated is in direct proportion to the amount of the chemical reaction, and since in the fish-tail burner all the carbon is not consumed, there is, therefore, in such a flame less heat to a given area than will be produced in the same area of a Bunsen burner.

This explains the use of the *blow-pipe*, which is a tube placed in a luminous flame and blowing through it from the lungs upon the flame a sufficient quantity of air to produce complete combustion. In the case of the blow-pipe, more heat is produced than in the Bunsen burner, for the reason that in blowing air into the flame the flame is

rendered much sharper and narrower than in a Bunsen burner, and in this way heat produced by chemical action is concentrated to a more limited area; hence by this instrument we are able to make a very intense heat, albeit in a very narrow area.

The **blow-pipe** consists of a bent and tapering brass tube ending in a very minute orifice, the larger end connecting with an appropriate mouth-piece which is pressed firmly against the lips when the air is

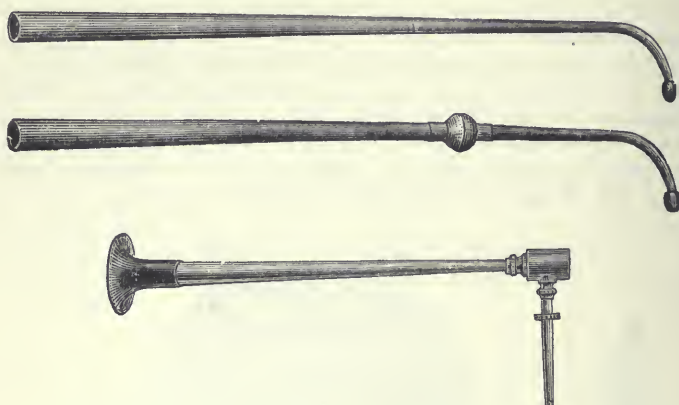


Fig. 40.—Blow-pipes.

energetically blown from the lungs through the pipe into the flame. In the cheaper forms of such blow-pipe, where no mouth-piece is provided, the larger end should be fitted with a piece of rubber tubing to prevent the lips coming in contact with the brass, which causes irritation. The continuous blowing of air through the blow-pipe is merely

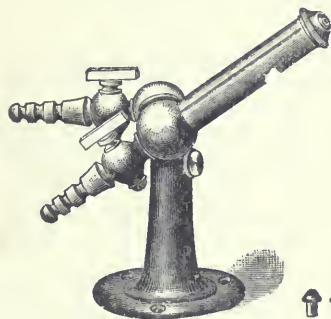


Fig. 41.—Blast lamp.

a matter of practice, the expert being able to inhale through his nose at the same time that he forces the air from his lungs through his mouth into the blow-pipe, and this must be done if a continuous flame is desired (Fig. 40).

The blow-pipe is used entirely for minute operations, such as small chemical work, or for insignificant cases of soldering in the jewelry business. For large operations in which a concentrated flame of high intensity is desired the blast lamp (Fig. 41) is used, and this is merely a modification of the blow-pipe in that the air is furnished by means of the bellows rather than by the lungs, and thus the instrument can be made larger.

In such a blast lamp a rather large flame of great intensity (1700°C.) can be easily attained, with the sole physical outlay of operating the bellows with the foot. When a very intense flame is desired, the same blast can be applied, but instead of illuminating gas, hydrogen is pumped in, and instead of air, oxygen is supplied. This forms the

oxyhydrogen blast lamp, and with this instrument a heat sufficient to melt platinum (about 2000°C.) can be attained.

Electricity is also used for the production of heat, and in future will play a more important rôle in our domestic economy. Even now electric stoves are used for warming purposes, while the greatest artificial heat is derived by the use of the Moissan electric furnace.

BATHS

Baths in pharmacy are devised for uniform distribution or for the limitation of heat. Thus the heat of a Bunsen burner, while very satisfactory, is apt to be more intense on one portion of the utensil being heated than on another, and to this cause is due most of the fractures on heating glass and porcelain utensils.

It is a dictum that, in heating substances in porcelain or glass utensils, the direct heat of a Bunsen burner should never be applied, but the flame should be uniformly distributed in the several ways at our disposal. The simplest form for the uniform distribution of heat is a sheet of asbestos or of tin, by which the cylindric flame of the Bunsen burner is spread out to the entire surface of the interposing medium, and a uniform heat is in turn transmitted from it to the container above.

Far more convenient than these, however, is a piece of gauze of iron or copper wire. Through such gauze the heat more readily penetrates than through asbestos, and at the same time the heat is as uniformly distributed by reason of the fact that the wire of which the gauze is constructed is an admirable conductor of heat, and the moment the flame comes in contact with the gauze, each of its many filaments aid in drawing the heat to every portion of the gauze. This has the effect of chilling the flame itself, hence such flame cannot pass through a wire gauze until the gauze has become heated to a temperature almost equal to the flame.

On this principle is based the construction of the Davy's safety lamp for use in mines, the use of which is of no pharmaceutic importance.

This phenomenon also explains why smoke cannot penetrate through such gauze, hence the value of gauze in the old form of gas-stove.

Sand-baths.—A sand-bath consists of a suitable flat dish in which is put a sufficient quantity of sand, the container of the liquid to be evaporated is placed therein, and the sand is piled around so as to completely surround it. Heat by means of the Bunsen burner or other flame is applied under the dish, and is gradually uniformly transmitted to all portions of the heating substance.

In this form of bath the heat is limited only to the heat produced by the burner, the sole aim being to distribute the heat uniformly.

Liquid Baths.—These consist of a suitable dish containing the liquid which is to be heated, the heat of which is in turn transmitted to the substance which it is desired to make hot.

The liquids used for this purpose are oil, glycerin, salt water, and water.

The *oil-bath*, like the sand-bath, uniformly transmits heat, but in addition thereto limits the amount of heat by reason of the fact that when the temperature of 260°C. is attained, the oil begins to burn; that is, undergoes decomposition, with the formation of the stifling

vapors of acrolein, which serve as a warning to the operator that a high heat has been attained.

Glycerin baths act on the same principle of operation as the oil-bath, and are limited for the same reason to 250° C. Both forms of baths are not so much used now, by reason of the fact that they do not really limit heat, and their method of warning to the operator is too disagreeable to be popular.

Toluene and *xylene baths* have, therefore, been constructed. (See Fig. 42.)

These liquids, boiling at 110° C. and 140° C. respectively, are placed in a jacketed vessel provided with a long glass tube, which acts as an upright condenser, causing the vapors to chill to a liquid and drop back into the container. In applying heat to such a bath the temperature rises to the boiling-point of the liquid, and the vapors, being condensed and returned to the container, are not lost; hence in such a bath we have not only the limit of temperature, but also a practically perpetual operation.

A *salt-water bath* is based on the same principle as the water-bath, save that it consists of an aqueous solution of some salt whereby the temperature of the boiling liquid is raised.

A bath made of salt water—saturated solution of sodium chloride—was formerly considerably used under the Latin name, “*balneum maris*.” Such a bath, however, possesses the great disadvantage of losing water by evaporation, with solidification of the remaining saline solution. It is, therefore, comparatively little used.

Water-baths.—In these the liquid to be used is water, and since water boils at 100° C., the temperature of the bath never rises above that point. A consideration of why water does not boil over 100° F. is left for a subsequent page, and, therefore, attention need be paid only to the various forms of water-bath (Fig. 43).

There is comparatively little necessity for buying expensive apparatus for use as a water-bath. Any tin pail filled with water in which the evaporating dish can be rested, or into which the flask can be sunk, can be used. An excellent form of expensive water-bath is a continuous steam apparatus shown in appended cut (Fig. 44). In this the water trickles from faucet into the well, *a*, the excess passing into the drain by overflow pipe *b*. A lighted Bunsen burner is placed beneath the well, and the small amount of water is rapidly converted into steam.

Steam Bath.—The steam bath differs from the water-bath inasmuch as the substance to be heated comes in contact with steam rather than with boiling water. While steam possesses the same temperature as boiling water (100° C.), it has a much larger actual quantity of heat, as will be explained under the subject of Latent Heat (p. 85).



Fig. 42.—Meyer's toluene bath.

Suffice it, therefore, to say that while in the steam bath, where the steam is brought into contact with the object to be heated at the ordinary atmospheric pressure, it will transmit to the same a temperature of $100^{\circ}\text{C}.$; on the other hand, if the apparatus is so arranged as to bring steam under pressure into the bath, a higher degree of heat can be attained. Thus, under the normal atmospheric pressure, 14.7 pounds to the square inch, steam has a temperature of $100^{\circ}\text{C}.$, while if steam is forced in until the pressure of 100 pounds to the square inch is obtained, said steam will have a temperature of $164^{\circ}\text{C}.$ Steam of any pressure above that of the normal atmosphere is said to be superheated. In large manufacturing concerns steam, either under normal pressure or in the superheated state, is practically the only form of heat used for the evaporation of delicate materials, as under such conditions there is little danger of decomposition of the product.



Fig. 43.—Copper water-bath.

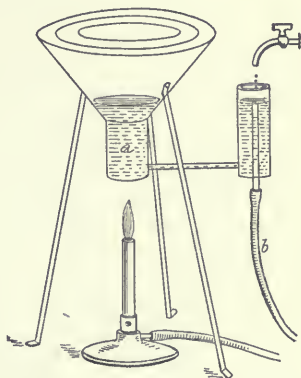


Fig. 44.—Meyer's water-bath

As steam baths have little practical application in retail pharmacy, a further consideration of the subject is omitted, the reader being referred to some text-book on advanced physics for information concerning laboratory steam kettles used for open pressure, and steam coils in which the steam can be heated under normal pressure or in superheated condition. A well-known variety of steam coil is the steam radiator, so largely used in heating houses.

The advantage a steam coil possesses from the manufacturing standpoint is that it can be placed directly into the kettle containing the substance to be cooked, and thus the substance can be directly heated, with practically no loss by radiation.

VALUATION OF HEAT

While we commonly speak of a thermometer as an instrument used to measure heat and cold, this definition is only partly true, as by means of the thermometer we measure the intensity of the heat, but not the actual quantity produced. Thus, while it is true that with a flame producing a temperature of $500^{\circ}\text{C}.$ we can accomplish more heating in a given time than we can with a flame of $150^{\circ}\text{C}.$, nevertheless, if we use a flame of $150^{\circ}\text{C}.$ for a sufficient length of time, we can usually accomplish as much as with a $500^{\circ}\text{C}.$ flame for a shorter time, although there are, of course, certain operations in which the intensity of heat is essential and a lower intensity would not do, even though applied for a proportionally longer time.

For measuring the actual quantity of heat the unit called the calorie has been devised. This unit represents the amount of work done by a flame.

A calorie represents the amount of heat necessary to raise one gramme of water from zero to one degree Centigrade, and will be spoken of more fully in the discussion of latent heat.

THERMOMETERS

Thermometers are instruments used for measuring the intensity of heat, but not the quantity thereof. Most of these instruments are based on the expansive power of certain liquids and gases. It is characteristic of a large number of varieties of matter to expand with a rise in temperature and, indeed, in regular proportion to the increase in temperature. Thus, an iron bar when heated expands, and if the expansion is limited to the longitudinal direction by encasing the bar in some way so as to prevent excessive lateral expansion, the expanded bar can be made to operate on a lever which moves a needle on a dial, and thus represents the intensity of heat, or the temperature. Such is the simplest form of the so-called *pyrometer*, which is intended to indicate temperatures greater than those shown by the ordinary form of thermometer.

To obtain an accurate thermometer the expanding substance should be liquid or gaseous, and in the early days such forms were devised, the first being by Galileo, who took a glass tube filled with alcohol and, as the heat was increased and the liquid expanded, it of necessity rose in the tube, and the rise could be measured, and in this way the rise in temperature was crudely estimated.

Similar rough instruments were devised from time to time, but the first step in the scientific direction was made by Fahrenheit between 1709 and 1724. Fahrenheit's first improvement was to use mercury in the thermometer instead of water or alcohol. The expansion of mercury is very delicate, and the density of this liquid being so much more than water, it would require a great deal less of same than of water or alcohol; hence in the Fahrenheit thermometer a much smaller tube was necessary than in the alcohol thermometer.

Fahrenheit also devised the first acceptable system of graduation, and this method, somewhat modified, is in use to this day. In graduating this thermometer Fahrenheit set about to find absolute zero, and finally decided that the greatest cold possible was that produced by a mixture of ice and salt. He prepared such a mixture, plunged his thermometer into same, and marked off the place where the mercury stood at this temperature, and denominated this point zero.

That the temperature of ice and salt is the greatest cold produced has proved false. It is usual, every winter, to find certain sections of the world where the temperature falls to as low as 50° or 60° below zero, while by chemical means the temperature of -251° C. has been attained, and experiments of physicists seem to point to the fact that absolute zero is -273° C.

Fahrenheit, however, having arranged his zero to his own satisfaction, next turned to look for another authoritative standard, and for this he determined upon the temperature of the blood. He, therefore, placed his thermometer under his arm, noted the point to which

the mercury rose, marked the space, and, for some reason not explained, denominated this "24." The space on the tube between zero and 24 he divided into 24 spaces called degrees, and his later experiments showed that on such a thermometer melting ice registered 8°.

Such a thermometer was intended for practical use, but it was soon found that the range of 24 between his zero and the temperature of the blood was scarcely sufficient for practical purposes, hence he devised a thermometer in which his former degrees were multiplied by four, and on such thermometer the freezing-point of ice was 32°, and the temperature of the blood 96°, and in terms of these degrees the boiling-point of water registered 212°.

Fahrenheit's thermometer, while a step in the right direction, has the misfortune of being based on an unscientific standard, as ice and salt do not produce the lowest known temperature, and, moreover, do not always represent exactly the same temperature experience has long since proved. In the same way the statement that 96° is the temperature of the blood is not absolutely accurate, inasmuch as the blood's temperature varies according to the condition of the person.

The graduation adopted by Réaumur in 1730 was a decided advance over the Fahrenheit system, inasmuch as Réaumur took for zero an absolute standard very easily determined (the melting-point of ice), and chose for his higher standard the equally accurate and easy unit of the temperature of boiling water. The latter, however, he denominated 80°, and the scale was, therefore, much improved by Celsius in 1740 in devising his Centigrade system, in which the melting-point of ice is zero and the boiling-point of water 100°, and the space between was accurately divided into 100 parts.

A comparison of the thermometers can be best shown by appended sketch (Fig. 45), in which it can be noted that while in the Fahrenheit thermometer the space between the freezing-point and boiling-point is divided into 180° (212°-32°), the same space on the Celsius is divided into 100°. Therefore, we can sum up the relative values as follows:

$$\begin{aligned} 180^{\circ} \text{ F.} &= 100^{\circ} \text{ C.} \\ 1.8^{\circ} \text{ F.} &= 1^{\circ} \text{ C.} \end{aligned}$$

To sum up:

Fahrenheit.....	Melting-point, 32°	Boiling-point, 212°	Difference, 180°
Centigrade.....	0°	100°	100°

which evolves the very important point that 1° Centigrade equals 1.8° Fahrenheit.

If both Fahrenheit and Centigrade had the same zero, or starting-point, the conversion of one into the other would be very simple, as it

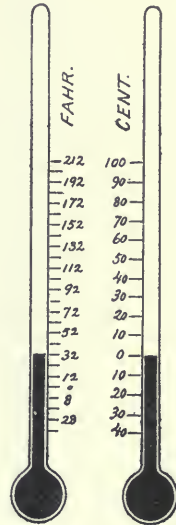


Fig. 45.—Comparison of Fahrenheit and Centigrade thermometers.

would only mean multiplying or dividing by 1.8, but where zero is on Celsius (or Centigrade) scale, 32° is on the Fahrenheit, and this 32, accordingly, comes in as a factor in changing one kind of degree into the other.

The rules of conversion can be abbreviated thus:

C. into F.: $\times 1.8$ and $+32$; F. into C.: -32 and $\div 1.8$, to which might be added for temperatures *below the freezing-point*, C. into F.: $\times 1.8$ and -32 ; F. into C.: $+32$ and $\div 1.8$.

To explain this, sketch a thermometer, similar to that pictured above, with Centigrade markings on one side and Fahrenheit on the other, beginning with a line marked 0 on the Centigrade side and 32 on the Fahrenheit.

Suppose we have 50° C. as a starter:

$50 \times 1.8 = 90.0 - 90$ *Fahrenheit degrees* but not *degrees Fahrenheit*. It means 90 Fahrenheit degrees above the melting-point of ice, and this point is 32° Fahrenheit degrees above the Fahrenheit zero. To get the actual *degrees Fahrenheit* in 50° C. we must add this 32 to the 90 already gotten, when we find that 122° F. $= 50^{\circ}$ C.

We will try the reverse problem: How many degrees Centigrade in 122° F.? We first get this figure on the same basis as zero Centigrade. This we do by subtracting 32 from 122, thus finding how many Fahrenheit degrees 122° is above the melting-point of ice. The difference between 32 and 122 is 90, and this, divided by 1.8, gives us 50. Therefore 122° F. equals 50° C.

Below the melting-point of ice, the matter is a little more difficult of explanation. Let us choose as our example how many degrees Fahrenheit in -35° C.

$35 \times 1.8 = 63$ *Fahrenheit degrees*, not *degrees Fahrenheit*, below the melting-point of ice—63 Fahrenheit degrees below the point that is marked zero on the Centigrade scale. The point marked zero on the Centigrade scale means 32° Fahrenheit; so the 63 degrees we have spoken of will be partly above and partly below zero Fahrenheit; 32 degrees of it will be above zero, the balance (63-32), which is 31° , will be below zero Fahrenheit, and thus we see that -35° C. is equal to -31° F. Inversely, if we want to know the value of -31° F. in the Centigrade scale, the first thing to do is to find how many Fahrenheit degrees there are between -31° and that point on the Fahrenheit thermometer which equals 0° C. This point is 32° F. If there are 31° below zero and 32° above that point to 0° C., then the number of Fahrenheit degrees between 31° F. and the point marked zero on the Centigrade scale is $31 + 32 = 63$.

In other words, -31° F. is 63 Fahrenheit degrees below zero Centigrade. Take 63, divide by 1.8, and we get 35, showing that -31° F. equals -35° C.

Conversion of Fahrenheit markings into Réaumur and Réaumur markings into Fahrenheit is of comparatively little importance in American pharmacy; therefore, a brief statement of the factor will suffice.

If 0.8° R. $= 1.8^{\circ}$ F.

Then, 1° R. $= \frac{1.8}{8} \times 1.8$, or 2.25° F.

Then to convert Réaumur into Fahrenheit multiply by 2.25 and add 32.

Then to convert Fahrenheit into Réaumur subtract 32 and divide by 2.25.

Varieties of Thermometers.—The usual form of thermometer consists of a glass bulb to which is attached a capillary tube. In it is placed a sufficient quantity of boiled mercury to fill the bulb and the lower portion of the tube. In the cheaper form of thermometer air is allowed to remain in the rest of the tube, which is sealed at the other end. In such thermometers, however, after continued use, the mercury is gradually oxidized by the air, rendering the instrument less accurate; therefore in the finer grades of thermometer the air is replaced by pure nitrogen, or a vacuum is made in the tube. This bulb and tube is then attached to a scale in the several ways that convenience suggests, the cheaper forms of thermometer being merely fastened to a piece of wood on which the scale is marked, while the chemical and pharmaceutical thermometers are usually inclosed in a glass tube inside of which the scale is placed, the intention in this case being to make the thermometer of cylindric form and sufficiently small to permit insertion through a cork, and then into the neck of a flask. On such thermometer the scale is made of paper or porcelain placed inside of the outer tube, or else it is engraved on the outer surface of the outer tube, the latter being particularly the case in the exceedingly small and delicate thermometers used in estimating boiling- and melting-points (Fig. 46).

In purchasing a thermometer for pharmaceutical purposes, always avoid those with paper scales, for should one be heated above 125° , it will scorch and even char, and the reduction in cost, as compared to the porcelain scaled thermometer, is too little to justify the economy.

It is needless to say that a Centigrade thermometer should be so accurately adjusted that the zero mark coincides with the height of the mercury column when plunged into melting ice, and that the 100° mark should agree with the height of the mercury column when placed in the temperature of steam.

To test thermometers for this purpose is a very simple matter. Such tests should be made from time to time on every thermometer used, as the glass in the thermometer, on aging, undergoes more or less contraction and therefore is apt to become inaccurate. Details of this testing will be found in Part VI.

A modification of the thermometer, sold in pharmacies, although not used in pharmaceutical operations, is the well-known fever thermometer. This is generally limited to the temperature of the human body—say from 90° to 110° F. Thermometers of such limited marking can, of course, be made smaller than those of larger scope, hence are more convenient for carrying by a physician.

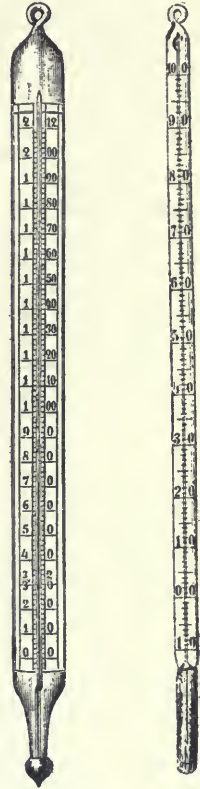


Fig. 46.—Chemical thermometers.

A popular form of this instrument is the so-called "self-registering thermometer," in which a portion of the mercury rises to the greatest height in the operation, and does not return with the rest of the mercury in contracting, thus leaving a register of the patient's temperature for examination by the physician on his return visit.

This break in the mercury column must be remedied before the thermometer is used for another operation, and this can be done very simply by grasping the thermometer by the upper end, raising same above the shoulder, and giving it a downward circular sweep. This, repeated several times, will remedy the break. The same treatment applies to a chemical thermometer when such a break in the filament occurs.

In some cases, however, the break is so persistent as to resist these efforts, when it can be brought down by gently warming the thermometer until the mercury rises to the broken part and forces same up, and then shaking the mercury down as above mentioned.

Other Varieties of Thermometers.—In pharmacy the thermometers above mentioned are practically the only ones employed, and to such thermometers the measure of the temperature is limited to minus 40° C., the freezing-point of mercury, and to plus 360° C., the boiling-point of mercury. In technic the measurement of higher temperatures is often desired, and for such purposes modifications called "pyrometers" are employed. The simplest form of pyrometer is the iron bar mentioned on p. 76. In another form the bulb and tube are made of finest porcelain, filled with air, which expands on heating, and the expansion is measured by the pressure of the air on a mercury column.

A more modern form of pyrometer is one in which electricity is employed, this acting on the principle that certain substances on heating show more resistance to electricity than when cold; indeed, in exact proportion to the increase of temperature. Therefore in such an instrument a current of electricity is conducted through the metal, which acts as the thermometer, and the temperature is estimated from the reading of the resistance gauge, or ammeter.

CHAPTER V

APPLICATIONS OF HEAT

In studying the dozen or more pharmaceutic processes involving the use of heat, we note that they can be best understood when presented in the following tabulated form:

I. Chemical change in the heated product:

- | | | |
|---|---|---|
| 1. Substance is inorganic, is strongly heated, and gives off gas. | { | (a) CO_2 eliminated, <i>calcination</i> .
(b) Gas eliminated with a flame, <i>ignition</i> .
(c) Gas eliminated with crackling sound, <i>deflagration</i> . |
| 2. Substance is organic. | { | (a) Strong heat without access of air, <i>carbonization</i> .
(b) Strong heat with access of air, <i>incineration</i> .
(c) Moderate heat only, <i>torrefaction</i> . |

II. Physical change produced in heated product:

1. Solid temporarily changed to liquid, *fusion*.
2. Solid or liquid temporarily changed to gas, *vaporization*.

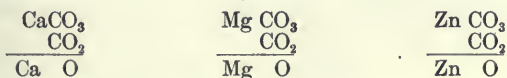
The modifications of vaporization can best be tabulated as follows:

METHOD.	OBJECT SOUGHT.	NAME OF PROCESS.
Simple vaporization.....	(a) Liquid residue.....	Evaporation.
	(b) Solid residue.....	Desiccation. Exsiccation. Granulation.
Vaporization and condensation.....	(a) Volatile liquid.....	Distillation.
	(b) Volatile solid.....	Sublimation.

Discussing the several processes individually we find the following points of interest and importance.

Calcination is the process of strongly heating a carbonate, whereby the gas, carbon dioxide, is driven off and the oxide of the metal remains.

Note the similarity between the words "calcination" and "calx." Calx is the Latin name for lime, calcium oxide, CaO , which is made by strongly heating calcium carbonate, CaCO_3 , when carbon dioxide, CO_2 , passes off. By the identical process—calcination—the oxides of magnesium and zinc are produced, as the following comparison shows:



The results of above subtraction—the remaining oxides—are said to be "calcined."

While the largest operations of calcination are performed in huge plants of masonry called lime-kilns, in the laboratory the substance to be calcined is usually placed in an evaporating dish (see p. 94) or in a crucible, the source of heat being a blast lamp.

Crucibles are utensils of various sizes, shapes, and materials, according to the objects for which intended. The name crucible dates from the middle ages, when the alchemists (the forerunners of the chemists), in invoking Divine aid in their operations, marked utensils of this character with the sign of the cross (Latin, "*crux*").

The modern crucibles are made of the following materials:

Fire-clay or Hessian crucibles (Fig. 47) are very cheap and convenient apparatus for conducting high heat experiments. They usually come nested, in sizes ranging from 15 to 500 Cc. Their chief drawback is that they are poorly annealed, and are therefore liable to break. Moreover, they are too porous to hold water.

Graphite crucibles are more expensive, but are durable. They are commonly called "black-lead crucibles," and are of the same material

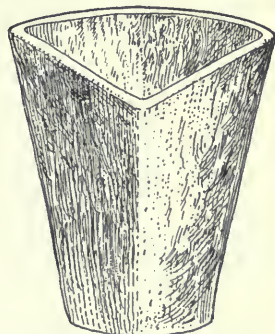


Fig. 47.—Hessian crucible.

of which the "lead" of pencils and the cake stove-blackening is composed. They are used more for chemical than for pharmaceutical work, as is also that form of crucible, "the cupel," which is made of bone-ash and used exclusively in metallurgy.

Porcelain crucibles are the most popular of all forms of crucibles, they being comparatively durable, easy to clean, and moderate in price. They can be obtained in all sizes, but the most convenient are those of 15 to 30 Cc. capacity (Fig. 48).

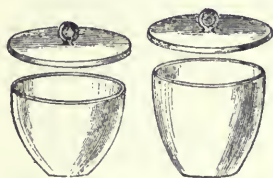


Fig. 48.—Porcelain crucibles.



Fig. 49.—Platinum crucible.



Fig. 50.—Crucible tongs.

Platinum crucibles are too expensive to be of ordinary pharmaceutical use, but the purchase of a platinum crucible is by no means a bad investment, as when the instrument has become worn out, the platinum of which it is composed can be sold at a price but little below its original cost. The most convenient size of platinum crucible is one of 10 to 15 Cc. capacity. They are of great value, because durable and because possessing powers of resistance against chemicals far greater than other metals. This great advantage must not be abused, however, for it should be clearly remembered that sulphides, hypophosphites, potassium and lead salts affect platinum, ruining the crucible in which they are heated.

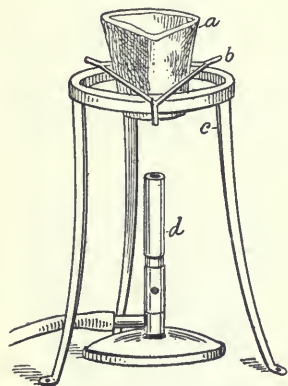


Fig. 51.—Heating crucible: *a*, Crucible; *b*, triangle; *c*, tripod; *d*, Bunsen burner.

Appliances necessary to the successful handling of crucibles are *crucible tongs* (Fig. 50), which are used for lifting crucibles and *triangles* on which they rest while being heated. These, as shown in cut, primarily consist of iron wire covered by clay piping, hence obtainable at small cost. The triangle rests on or is supported by a tripod (Fig. 51) or else on the ring of a retort stand.

Ignition is the process of heating inorganic matter strongly, whereby certain portions are driven off in gaseous forms (usually with formation of a flame), leaving the non-volatile residue. This process is used in pharmacy in some analyses, for example: the United States Pharmacopœia of 1890 directed in a test for sulphurated antimony, that the same, on being heated, should lose its sulphur by ignition, leaving a residue consisting chiefly of antimony oxide. Likewise ignition is

used in the burning of iron pyrites, when the sulphur which it contains combines with the oxygen of the air to form the gas sulphurous oxide, leaving a residue (ferric oxide) behind.

The ignitions of pharmacy are usually performed on platinum foil, in crucibles, or in evaporating dishes, or even on the end of a spatula or knife-blade. Thus the presence of calcium sulphate in a sample of precipitated sulphur is easily detected by igniting a bit of the suspected chemical on the blade of a spatula, when it leaves a residue only in case calcium sulphate is present.

Deflagration is the process of strongly heating a substance readily yielding oxygen, when this element is evolved with a characteristic crackling sound. This phenomenon occurs when potassium nitrate or potassium chlorate is heated, and the process is quite different from that of "decrepitation," which will be explained in the chapter on Crystallization.

Deflagration is used in no process of pharmacy, but it is of value in the testing of some official chemicals, such as potassium nitrate. To produce deflagration the salt is sprinkled on hot coals, or is heated on platinum foil or is placed in a deflagrating spoon (Fig. 52).

Carbonization is the process of strongly heating an organic substance (that is, a substance containing carbon) in a vessel protected from access of air. Under this treatment the hydrogen and oxygen usually found in organic substances associated with carbon pass off in the form of water, leaving behind the carbon or charcoal.

This process is rarely employed in pharmacy, and when utilized may be performed in a rather tightly covered crucible. The typical example of carbonization is the manufacture of charcoal,—“the charcoal burning,”—an industry of the southern United States.

If carbonization is carried on in a retort and the volatile products collected, the process is called *destructive distillation*. By such destructive distillation of wood, wood-alcohol, acetone, and acetic acid are now made.

Incineration is the process of strongly heating an organic substance with access of air, whereupon the carbon, as well as the hydrogen and oxygen, is dissipated, as oxidation products, and nothing is left but inorganic salts found in the original substance, and commonly called *ashes*.

Incineration is like carbonization, save that the former process is performed in a utensil where the air cannot get at the organic substance, while in incineration the air has full access. This makes the important difference that in carbonization the residue, after heating, is carbon or charcoal and inorganic salts; while in incineration the carbon combines with the oxygen of the air to form the gas, carbon dioxide, CO_2 , which passes off, leaving behind only a small residue (ashes) consisting of inorganic salts. Such an operation is every open fire; such is the process of cremation. Its use in modern pharmacy is limited to chemical examinations, the amount of ash yielded by drugs being in some cases a valuable criterion of quality. Such ash estimations are performed by carefully drying a crucible by heating in Bunsen burner for fifteen



Fig. 52.—
Deflagration
spoon.

minutes, weighing same after cooling in a desiccator (p. 109), carefully placing the drug in the crucible, and weighing, incinerating same, and finally weighing the remaining ash.

In alchemistic times the ashes from certain plants were supposed to be of great value in medicine. Thus the ashes of wormwood, which modern chemistry has shown to consist chiefly of potassium carbonate, was highly esteemed under the name of "*sal absinthii*," or "*salt of wormwood*," two synonyms which still cling to the commercial salt.

Torrefaction is the process of more moderately heating organic matter with access of air, in which case the active principles of the substance are not eliminated, but are merely modified. The process is what is commonly called "*roasting*," and a familiar illustration is the roasting of coffee, peanuts, etc. In pharmacy it is an obsolete process, although formerly "*torrefied rhubarb*" enjoyed considerable vogue.

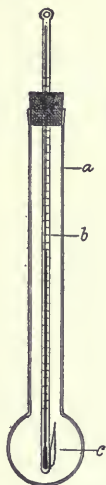
The operation is performed by placing the drug in an evaporating dish, watch-glass, or crucible, and exposing it to a steady moderate heat; best applied by means of a drying oven (p. 109).

Fusion is the process of converting a solid into a liquid by means of heat without the aid of a solvent, the object usually being to enable the straining of the fluid mass or the molding of it to some special shape at the moment of solidification. The process is so well known that it is scarcely necessary to do more than refer to the well-known operation of melting wax as an illustration of fusion. This, therefore, is an official fused substance, as is also the official molded silver nitrate.

Substances are fused in any appropriate vessel, —crucible, test-tube, or dish,—and the melted mass strained or poured into molds as desired, it being hardly necessary to state that since the liquid returns to solid form as soon as the heated mass reverts to ordinary temperature, the fused condition is merely temporary.

The *melting-point* of a substance is estimated by placing the powdered mass in a very small tube, attaching this tube to a thermometer, and immersing the thermometer and tube into concentrated sulphuric acid contained in a beaker or flask (Fig. 53). Heat is then applied to the acid, and the moment the substance fuses, the thermometer is read.

Fig. 53.—Melting-point apparatus: *a*, Flask; *b*, thermometer; *c*, substance in tube.



Vaporization is a general term meaning the change of a solid or a liquid into a vapor; the latter term meaning the gaseous form of a substance, which at ordinary temperatures is either a solid or a liquid.

Liquids and solids are held together by the force of cohesion exerted by their molecules. In gases, the molecules, instead of being held together, actually repel each other; the force of cohesion is practically overcome. To overcome this requires much energy, and it is into such energy that that surplus heat of vaporization is converted. Steam, when placed in a position in which it can gradually assume normal temperature, reconverts the energy necessary to keep its molecules

apart into heat, imparts this heat to the surrounding media, and returns to liquid form. Even so do all volatile liquids, and the process whereby a vapor passes into a liquid form is called *condensation*. This condensation is usually hastened, in general practice, by application of cold water or ice to the vapor.

The form of energy necessary to keep the molecules of a substance sufficiently far apart to maintain the vapor form of that substance is called "*latent heat of vaporization*." Latent heat can be simply explained as follows: We take a flame that would register 500°C . on a thermometer and put over it a pot of hot water; the temperature of that water will never rise much above 100°C . Possibly it may reach 102° or 103°F ., but hardly higher. What then becomes of the surplus heat of the flame? It becomes hidden, it is said; hence the term "latent," from the Latin "*lateo*," to hide.

But the term is scarcely correct. We learned in Chapter IV. that heat was molecular motion—a kind of energy. Now this energy can be transformed into other forms of energy under certain conditions, and that is what happens in the case under consideration.

Water when it reaches 100°C . is converted into steam—it is changed from a liquid to a gas. This transformation means work. It means expenditure of energy, and the energy needed comes from those 400°C . of heat which we say becomes latent.

One of the fundamental rules of physics is that energy is never lost. We may lose sight of it, but it merely assumes another form. Accordingly, all that energy—all those 400° latent heat in the flame—is to be found in the steam, ready for other work, as can be shown as follows:

Take equal parts of water at 100° and at 0° and the mixture will have a temperature of 50° , the mean of 100 and 0. Take, on the other hand, equal parts of steam at 100° and water at 0° and we get water at 100° . This shows that steam at 100° contains more heat than water at 100° ; that this extra heat is used to hold the molecules of the vaporized water apart. The exact quantity of energy has been gaged, and it is found by experiment that one part of steam at 100° is not merely capable of raising one part of water from zero to 100° , but can actually raise 5.4 pounds of water from zero to 100° . Accordingly, 1 pound steam can raise 540 pounds water 1°C ., and we, therefore, say the latent heat of steam is 540 calories (the exact figure is 535.9).

This explains why steam is so much more valuable for heating purposes than an equivalent amount of water at the same temperature, as it also explains why steam is so much "hotter" than water at the same temperature; why a steam scald is so much more painful than a scald from boiling water.

The changing of the liquid into vapor occurs at all temperatures. That water, for example, evaporates freely at ordinary temperatures is demonstrated by the fact that a towel soaked in water and hung in such a position as to allow access of air will readily dry, or, in other words, water will evaporate therefrom. However, water evaporates with the greatest facility only when the boiling-point is attained.

The **boiling-point** is of such pharmaceutic importance that it behoves every student clearly to understand when and how *ebullition*, or boiling, commences.

When a pot of water is on the fire, the first part of the liquid to reach 100°C . is that nearest the flame—at the bottom of the pot. It is, therefore, the water at the bottom that is first changed to steam.

Steam is vapor—a state of aggregation in which the molecules tend to get away from each other. They have overcome the force of cohesion, are wild to break bounds and escape into the open air. This tendency of a liquid to expand to a gas is called the *tension of its vapor*.

The steam at the bottom of the kettle has its tension, but it is restrained in two ways. It has above it the weight of the column of water in the pot, and on top of this the weight of the atmosphere, which presses down with the force of almost fifteen pounds to each square inch of surface. Before the steam can escape, before the bubbles of vapor can rise to the surface, the combined restraining power must be overcome by the tension of the vapor of water, and the point where the tension of the vapor overcomes the pressure of the atmosphere is the boiling-point, and is attained only at a certain degree of heat absolutely fixed for each liquid.

Vapor may pass off at temperature under the boiling-point, but it then passes from the surface of the liquid, with little or no disturbance, and takes place rather slowly.

It is only at that supreme moment—that moment when the heat attained is sufficient to raise the tension of the vapor above the pressure of the atmosphere, that the liquid boils; that bubbles of steam arise from the bottom of the heated liquid.

The statement that a liquid begins to boil when the tension of its vapor is greater than the pressure of the atmosphere can be easily demonstrated by the following simple experiment:

A glass tube sealed at one end and about three feet long is exactly filled with mercury, and inverted into a bowl containing the same liquid. It will be noted that the mercury in the bowl will support a column of mercury inside the tube, usually about thirty inches above its surface. The force supporting the column of mercury thirty inches high is none other than the pressure of the atmosphere on the surface of the mercury in the bowl, and if the area of the interior of the tube be one square inch, it will be found that the weight of this thirty-inch column of mercury will be 14.7 pounds.

From this we deduce the statement that the usual atmospheric pressure is 14.7 pounds to the square inch, and this has been proved in other ways.

We thus learn what is meant by the pressure of the atmosphere. Now, in order to demonstrate the second part of the statement just given above, that a liquid begins to boil when the tension of its vapor overcomes the pressure of the atmosphere, we insert into the vacuum in the tube above the column of mercury an appropriate quantity of water, which at ordinary temperatures will have but little effect on the height of the mercury column. The moment we begin heating the water a change occurs, the mercury being gradually pushed down toward the mercury in the bowl by the expansive force of the warmed vaporized water; in other words, by the tension of the vapor of the water. As the heat is continued the pressure of the vapor of the water pushes the mercury farther and farther down; in other words, gradually

overcomes the pressure of the atmosphere, and this before the liquid begins to boil.

The moment the liquid begins to boil it is observed that the mercury has been completely forced from its former place, that is, the column of mercury thirty inches high has disappeared—the tension of the vapor of water has become equal to the pressure of the atmosphere.

This experiment can be carried on with liquids other than water, in which cases, with equal success, it being found that the column of mercury is completely forced down from the barometer at the moment the boiling-point of that liquid is reached.

That different liquids possess different boiling-points is, of course, too well known to require much comment, but mention may be made that while water boils at 100°C ., alcohol boils at 78° , chloroform at 60° – 61° , and ether at 37° .

Estimation of boiling-point is discussed in Part VI. and on p. 95.

As shown in the table given on p. 81, there are several modifications of vaporization, all of which will now be discussed.

Evaporation, in the strict sense of the word, means the vaporization of a liquid from a liquid, as, for example, the removal of water from glycerin by gentle heat. In a broader sense of the term, however, it includes the removal of liquid by heat from any solution. Thus it is more usual to speak of evaporating a solution of salt than of evaporating water from glycerin.

Conditions Affecting Evaporation.—The rapidity of the evaporation depends rather largely on the following conditions:

First, *amount of heat*.

The greater the source of heat, the more rapidly the evaporation will take place.

Second, *the amount of atmospheric pressure*.

As noted above, the less the atmospheric pressure, the lower the temperature necessary to bring the liquid to the state of ebullition. Thus, while water boils at the ordinary atmospheric pressure of 14.7 pounds to the square inch, at 100°C ., the same water, placed in a container from above which the pressure of the atmosphere has been lessened to only 1.7 pounds to the square inch, will boil at 50°C ., and, on the other hand, the same water confined in a container in such way that the pressure on the surface of the liquid is 100 pounds to the square inch, will not boil until the temperature of 164°C . is reached.

Therefore, many cases of evaporation are carried on in special apparatus in which the atmospheric pressure has been lessened, such apparatus being called vacuum pans.

Third, *amount of humidity in the air*.

An aqueous solution, on evaporating, is converted into steam which, of course, passes off into the atmosphere, and the rapidity with which the air receives this vapor is in proportion to the amount of moisture already contained in the atmosphere. A dry air naturally absorbs more moisture than will a humid atmosphere, hence liquids evaporate more rapidly in dry air than in humid air.

Fourth, *character of the solvent*.

In evaporating liquids at the boiling-point, the nature of the solvent influences to a greater or less degree the rapidity of evaporation: a thin mobile liquid usually evaporates more rapidly than a dense and viscid liquid. The question of evaporation is not, however, a mere question of relative specific gravities, as is shown by the fact that the very volatile chloroform (boiling-point, 60° – 61°) is one and one-half times heavier than water, the volatility of the chloroform being due to the mobility of the liquid and not to its density. The reason a mobile liquid evaporates more readily than does a viscid one is because its molecules, having little adhesion, freely separate from one another, thus affording the exit of the vaporized portion. In the case of viscid liquids, like syrup or mucilage of gum arabic, the molecules do not freely separate from one another, and on evaporation of such liquids this fact can be noted by examination of the bubbles formed as the liquid vaporizes.

The influences affecting evaporation by boiling and evaporation below the boiling-point are of different character, in the first case the main factor being the extent of surface exposed to the heat, while in the latter case it is the extent of surface exposed to the air; hence each of these can best be considered separately.

Evaporation by Boiling.—Since heat is necessary to facilitate boiling, it follows that the greater the surface exposed to the source of heat, the more rapidly the liquid will boil, and this result is obtained not simply because of such broadening of the surface exposed to the heat, but also by the fact that such broadening implies a corresponding decrease in the depths of the liquid. As an illustration, the volume of 100 Cc. water will have much less depth if poured into a flat dish than if retained in a graduate, and that the lessening of the depth facilitates ebullition can be understood when the fact is noted that the liquid begins to boil not merely when the tension of its vapor is greater than the pressure of the atmosphere, but when such tension is greater than the pressure of the atmosphere plus the pressure of the column of the liquid above. Hence in evaporating at boiling we try to place the liquid in as thin a layer as possible, by choosing flat forms of evaporating dishes and the like.

Since evaporation by boiling is influenced by the amount of surface exposed to the heat, the character of the vessel plays its part, as certain materials and forms transmit heat better than others. Thus iron and copper vessels are better for evaporation of liquids than are porcelain, because of the greater conductivity of the metals. This statement, however, does not mean an unqualified recommendation of iron and

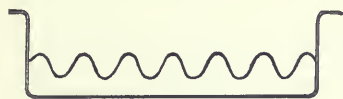


Fig. 54.—Corrugated pan bottom.

copper utensils for evaporation of pharmaceutical products, as the question of preserving the purity of the chemicals is of far more importance than is the rapidity with which it can be evaporated, and metallic vessels are very apt to contaminate the products.

As to form, a corrugated vessel will transmit heat more readily than a flat-bottomed one, as is self-evident by examination of Fig. 54, where it is seen that a corrugated vessel actually exposes about twice as much surface to the heat as a flat-bottomed vessel of the same diameter.

In the same way a liquid will evaporate more readily in a rough vessel than in a very smooth one. For this reason in boiling water for domestic purposes a rough iron kettle is preferred, as the roughness of the utensil acts the same as does the corrugated bottom of the typical evaporating vessel mentioned above. The external roughness and lack of luster of the vessel is also a contributing factor, inasmuch as finely polished external surfaces act as reflectors of heat as well as light, actually throwing off the heat instead of transmitting it to the interior; hence, the making of boilers and kettles with rough and dark exteriors.

Evaporation Below the Boiling-point.—While in this case of evaporation more attention is paid to the surface exposed to the air than to the surface exposed to the heat, the two usually resolve into the same thing, because in obtaining the largest surface exposed to

the air we use a shallow vessel, even as we do in attempting to increase the surface exposed to the heat.

The pharmaceutical application of evaporation below the boiling-point is generally limited to the desiccation of substances and the evaporation of liquids which are apt to be decomposed when heated on a wire gauze, and, therefore, directed to be heated on a water-bath. While a water-bath has itself the temperature of 100°C ., it never transmits that heat to the substance placed on same. The actual temperature of a liquid heated on a water-bath is usually from 90° to 95°C ., the surrounding air causing that much loss of heat when transmitted from the water-bath to the substance. For this reason an aqueous liquid cannot be made to boil on an ordinary water-bath and, therefore, the evaporation of such aqueous liquid on a water-bath is rather a tedious matter. The evaporation can be facilitated by constant stirring, this stirring producing the same effect on the liquid as the corrugated bottom of the evaporating dish mentioned above, by increasing the extent of surface exposed to the heat. Stirring the liquid produces waves thereon, and as a wave in its ideal sense represents a corrugation, it means an increased surface exposed to the air. To attempt to evaporate an aqueous solution on a water-bath without constant stirring is practically impossible.

In some cases liquids are evaporated without recourse to any ordinary heat, that is, at the ordinary temperature, and such is called spontaneous evaporation.

Spontaneous Evaporation.—An illustration of this is seen in the drying of clothes when the same are spread to their utmost extent and suspended from lines, thus increasing the surface exposed to the air. Applications of this principle are comparatively limited in pharmacy, as in most cases the ordinary sources of heat are generally cheap enough to permit their use in evaporation.

Two cases, however, in which spontaneous evaporation is used commercially may be cited. In the first case, in Germany, salt is extracted from mineral waters by this process with profit, where evaporation with ordinary fuel would not pay. In such process the water is allowed slowly to trickle through wooden frames about thirty feet high and many feet long, filled with straw and twigs. This framework has no sides, thus affording a thorough circulation of the air, and as the liquid trickles through the frame, enough of the water is evaporated to cause it to reach the bottom trough sufficiently concentrated to justify using heat for the final evaporation.

Another case of spontaneous evaporation, applied commercially, is the evaporation of sea-water practised in Portugal and Spain, where direct solar heat is utilized for evaporation.

In this case the solution is pumped into shallow ponds and allowed to stand exposed to the sun's rays for three or four weeks, when sufficient evaporation will have occurred to start a crystallization of the salts.

Evaporating Apparatus.—The articles essential to evaporation in pharmacy are dishes and stirring rods and the appropriate sources of heat. Other apparatus used in the technic and of advantage in pharmacy, though not essential, are fume closets and hoods, grommets, and the very useful but expensive vacuum apparatus.

Evaporating Dishes.—These are made of porcelain, glass, or platinum, shallow vessels of the appearance shown in Fig. 55. For practical purposes the porcelain dish is generally preferred, being more durable and comparatively inexpensive. Glass evaporating dishes are too fragile for continued use, and are chiefly used in crystallization. Plati-

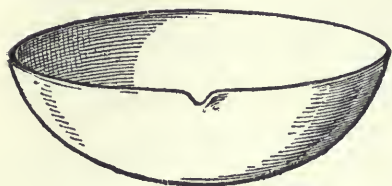


Fig. 55.—Evaporating dish.



Fig. 56.—Evaporating dish, graduated inside.

num evaporating dishes are used in some chemical operations, their expense precluding general use.

In some cases of evaporating directions are given to “reduce” the liquid “to a certain bulk,” and for this purpose graduated evaporating dishes have been introduced (Fig. 56).

Instead of same, however, a home-made contrivance answers just as well, and this consists of a rod or cylinder of wood, measuring the volume by marking same on the stick (Fig. 57).

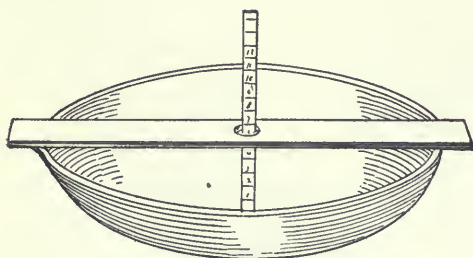


Fig. 57.—Evaporating dish, graduated with stick.

To avoid the necessity for these graduated evaporating dishes the United States Pharmacopœia directs such evaporations to be conducted until the liquid is brought to a definite weight, in which case the evaporating dish is transferred from the source of heat to the scales. Of

course, in order to learn the weight of the liquid the weight of the evaporating dish itself, “the tare,” must have been previously ascertained. It is a good practice to tare each dish when obtained, and mark the tare in lead-pencil on the outside.

An important point to remember in the use of an evaporating dish is that same should never be heated in direct flame, the source of heat being modified either by use of the wire gauze or by disks of asbestos.

A warm evaporating dish should never be placed directly on a counter. If the counter is of wood, the heat may be sufficient to soften the varnish, and if it is of metal or marble, the rapid chilling of the evaporating dish, through the difference in its temperature and that of the surface of the counter, may cause a fracture of the dish. It should, therefore, be placed on a mat, or, better, on a “grommet,” the latter consisting of a rubber ring the construction of which is shown in appended sketch (Fig. 58).

Stirrers.—The stirring of an evaporating liquid is a valuable aid to rapid evaporation; hence the use of stirrers. Their construction depends on the uses for which they are intended. Thus, in large operations, such as evaporating infusions of drugs containing no acid, a large tin spoon is as valuable as a stirrer. In other large operations, such as making syrups, a wooden paddle answers. For the more delicate operations of pharmacy and chemistry glass rods are required, which can be obtained in various diameters from chemical

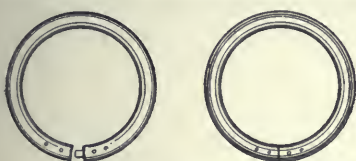


Fig. 58.—Grommet.

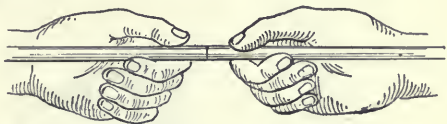


Fig. 59.—Breaking glass rod.

apparatus manufacturers in three- or four-foot lengths, and then can be readily broken by scratching with a file and applying pressure on either side of the scratch, thus fracturing same, as shown in Fig. 59.

In cutting these stirring rods it is best to make them of such length that about two-thirds will rest within the container for which intended.

If more than a third projects over the outside, they are apt to be top heavy. After breaking the rod to the appropriate length, the rough edge should always be smoothed by rotating in a Bunsen flame until slight fusion is effected. If the rough edges are permitted to remain, there is danger of scratching the interior of the evaporating dish.

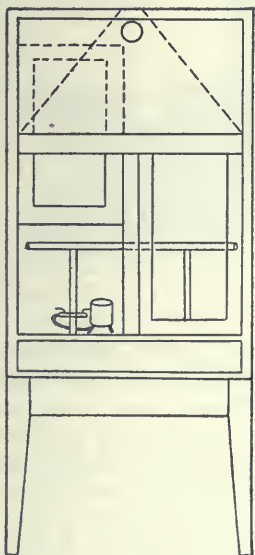


Fig. 60.—Fume-closet.

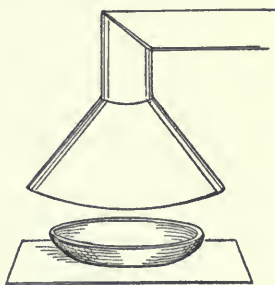


Fig. 61.—Hood.

Fume-closets and Hoods.—Evaporation of a large quantity of liquids, and particularly the evaporation of liquids yielding disagreeable or corrosive vapors, in a closed room is objectionable, and to carry off these vapors fume closets and hoods have been devised. The principle of these two is the same, both being an inclosed space connected with a flue or chimney, through which there is sufficient current of air to draw off the offensive vapors. The closet is a large piece of

furniture constructed of wood and glass, and attached directly to the wall (Fig. 60). For a hood can be utilized appropriate lengths of stovepipe, the lower end of which is arranged around a metallic dome, directly under which is placed the evaporating liquid. Hoods work best if connected with a suction pump (Fig. 61).

Vacuum Apparatus.—The principle of the operation of a vacuum apparatus having been explained, it is necessary here merely to state that many organic substances, when evaporated even at water-bath temperature, will dissociate. Thus, if sugar is evaporated even at the



Fig. 62.—Stokes' vacuum still.

heat of 100°C ., the brown substance, molasses, is produced with accompanying vitiation of the finished product. For all such cases vacuum evaporation is desirable, and this is performed in a suitable evaporating utensil, usually heated by the steam coil, and connected with a dome apparatus which, in turn, is connected with an appropriate vacuum pump (Fig. 62). From the illustration it will be seen that the vacuum pan is connected with a worm condenser (p. 97), thus permitting the recovery of the solvent, if it is costly. This is usually the case when vacuum evaporation is employed in pharmaceutical manufacturing, where it is chiefly used in the concentration of alcoholic percolates, such as in making extracts (p. 276). As seen in the illustration, the air is exhausted by means of a pump operated by steam-power. In the technic such evaporating apparatus is largely used, and for pharmaceutical and chemical purposes a small and comparatively inexpensive apparatus of the same character has been devised (Fig. 63).

For removing atmospheric pressure, any form of air-pump can be used, but usually in laboratory work the water suction pump (Fig. 64)

is employed. This can be attached to the ordinary hydrant, provided the water pressure is sufficient. In this simple and useful apparatus the rapid flow of the water through a narrow point draws with it the air from the tube and from the connected aspirator, and if the water pressure is sufficient, a vacuum of 17 mm. can be readily obtained. By a vacuum of 17 mm. is meant that the height of a column of mercury supported by the atmosphere is reduced from 760 mm. to 17 mm.; the latter figure, therefore, showing a pressure of only $\frac{17}{760}$ of the ordinary atmospheric pressure. The indication of this pressure is accomplished

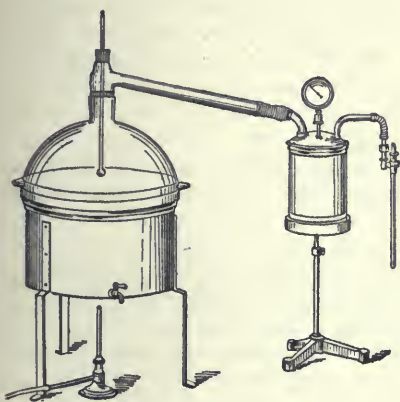


Fig. 63.—Pharmaceutic vacuum pan.

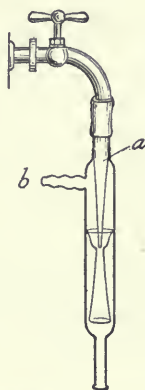


Fig. 64.—Water suction pump: *a*, Water-tube; *b*, air-tube.

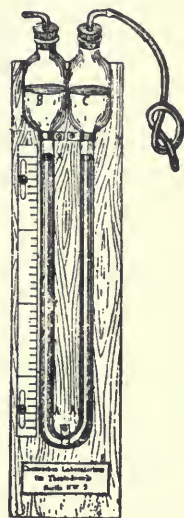


Fig. 65.—Manometer.

by the instrument called the manometer. Some forms of manometer are delicate apparatus made on the principle of the steam gage, but for chemical work a simple manometer consisting of a bent tube (Fig. 65) filled with mercury, and set up against a scale, answers all purposes.

The simple form of such vacuum evaporating apparatus is that figured in vacuum distillation (p. 104).

DISTILLATION

Distillation is the process of converting a liquid into a gas, and condensing the gas back into a liquid.

Objects.—In distillation we have an admirable method of purifying volatile substances. If such a liquid contains impurities less volatile than itself, the impure product is placed in a still and heated, whereupon the substance desired will distil, leaving the less volatile substance behind. In many cases the foreign matter is water, in which case distillation is of great value in strengthening the product under consideration. This is particularly the case in a separation of alcohol from whisky or other alcoholic liquors, which is always accomplished by distillation. The liquid thus purified by distillation is called a "*distillate*."

In other words, distillation is evaporation of a liquid and subsequent condensation of its vapor. Evaporation has been described

on a preceding page, and here it is necessary to turn attention only to the second part of the process, namely, condensation.

As already learned, to convert a liquid into a vapor means the expenditure of considerable energy, which energy is obtained from a source of heat, this heat being converted into the other form of energy,—that necessary to keep the molecules of the gas separate from one another,—is no longer indicated by means of the thermometer, and such heat is called “latent.” If the converting of the liquid into a vapor means the expenditure of considerable heat, so in the inverse operation of changing the vapor back into a liquid an equal amount of heat is given off. In other words, heat is rendered latent in converting a solid into a liquid or a liquid into a gas, and, in turn, latent heat is rendered sensible by changing gas back to liquid, or liquid back to a solid form. Therefore to condense a vapor back to liquid form the chilling of the vapor is necessary, that is, the latent heat must be absorbed by being brought into contact with a cooling agent. The converting of the vapor back to the liquid form in condensing can be accomplished in many cases by bringing the vapor in contact with chilled water.

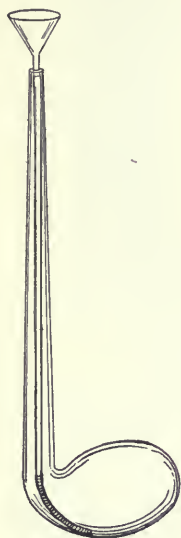


Fig. 66.—Charging plain retort.

The amount of water required can be figured out mathematically, and depends on the temperature of the vapor and the temperature of the water before and after its use as a condensing agent. Thus, 1 kilo of steam at 100°C. will take 23.2 kilos of water at 20°C. for condensation, and the temperature of the water after condensation will be 45°C. , or 1 kilo of steam at 100°C. will take 24 kilos of water at 10°C. , heating it to 35°C. during condensation.

The apparatus required for distillation consist primarily of a *boiler*, in which the liquid is vaporized, and the *condenser*, in which the vapors are chilled until they are condensed to the liquid form. This combination of boiler and condenser is commonly called the *still*, and the many forms of this apparatus which are in use can be roughly divided into two classes—the alembic form and the retort form.

In the alembic form the condensing apparatus is directly over the boiler, while in the retort form the condensing apparatus is placed on one side of the boiler, thus not directly over the source of heat. The retort form of a still being more commonly used, it will be discussed first.

Retort Still.—So called because the original form of boiler for such operation was the glass instrument called a retort, which consisted of a glass flask with a long and tapering neck, bent at an acute angle. This simple form of retort is inconvenient by reason of the difficulty in filling such a retort, it being, of course, essential to secure a pure distilled product that the neck of the retort through which the distilling vapor passed should be free from impurities.

Therefore, the charging of such a retort must never be done by pouring the liquid down the neck, but the liquid must be introduced into the bowl of the retort by means of a funnel with a long tube connected therewith (Fig. 66).

Of greater convenience is the tubulated retort, the use of which is strongly urged in preference to the plain form. In purchasing such a retort be sure, however, that the tubulure is correctly placed, namely, directly over the bottom of the bowl, for, if the tubulure is placed otherwise, there is danger of spattering the neck of the retort with the impure liquid when it is poured in (Fig. 67).

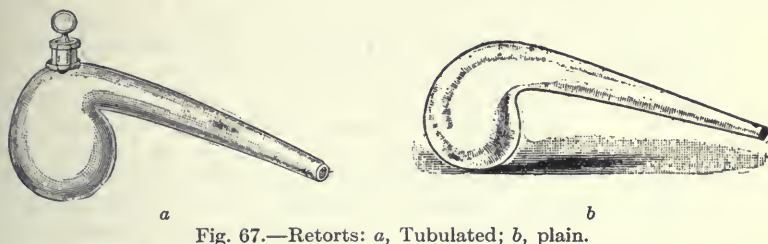


Fig. 67.—Retorts: *a*, Tubulated; *b*, plain.

For the purpose of insuring the cleanliness of the neck of the retort care should be taken to select such having an acute bend. In retorts where the bend is round rather than acute, there is always danger of some of the liquid being drawn over the bend by capillary attraction into the neck of the retort. Retorts formerly had extended use, but at present they are in less demand and have been largely replaced by the more convenient and simple instrument, the distilling flask (Fig. 68), which consists of a chemical flask of required size, provided

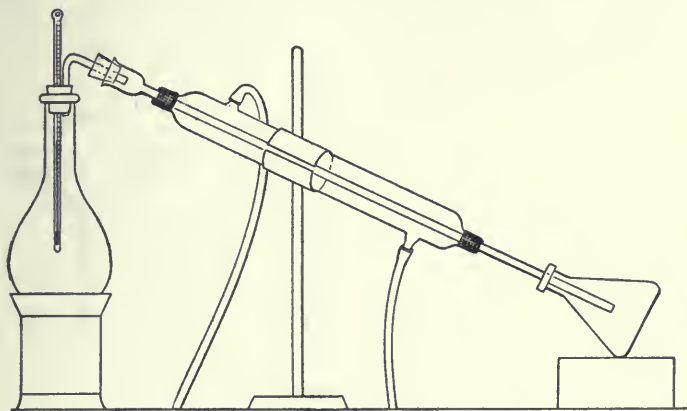


Fig. 68.—Flask distillation.

with a cork stopper through an orifice of which passes a piece of bent glass tubing. In many cases a second hole is bored through the cork for the insertion of a thermometer. Details of fitting the connections will be given later. A more compact form of distilling flask for small operations is shown in the illustration here given (Fig. 69).

Condensers.—The condenser is the part of the apparatus in which the vapors are converted into a liquid, and consists of an appropriate container which can be kept cooled by means of ice or running water.

The simplest form of condenser is a *receiver*, which, in many cases, consists of a chemical flask resting within a basin, in which water or ice can be placed. The more elaborate forms of receivers, the tubulated and quilled, are shown in Fig. 70.

The receiver is the only form of condenser necessary for liquids boiling above 150°C. , in which case the temperature of the atmosphere is sufficiently below the temperature of the boiling liquid to permit



Fig. 69.—Glass-stoppered distilling flask.

its use for condensing purposes. In most cases, however, it is usual to have the receiver immersed in water, as mentioned above. In distilling liquids boiling below 150°C. , such as water or alcohol, the air is not sufficiently cool to act as a perfect condenser, and in such cases cold water is usually substituted, it being convenient to arrange a continuous stream of water by connecting the condenser with a hydrant. In cases where a hydrant is not convenient, a stream of running water can be maintained by placing the water in a container several feet above the condenser, and maintaining a continuous stream over the condenser by

means of the syphon. In such cases of water condensation, preference is usually given to the Liebig condenser.

Liebig Condenser.—This consists of a long glass tube which is connected with the boiler, be it a retort or flask, thus affording passage to the vapor arising from the boiler. Surrounding this glass tube is a second tube, either of glass or metal, through which passes a stream of cold water (Fig. 71).

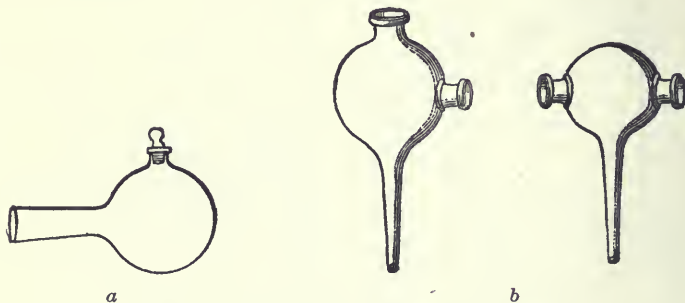


Fig. 70.—Receivers: *a*, Tubulated; *b*, quilled.

In the typical form of Liebig condenser the outer glass tube has soldered thereto ingress and exit tubes, and both ends of the cylinder are tapered so that the joint between the inner glass tubes and the condensing cylinder is made water-tight by means of rubber tubing.

Such condenser, while handsome, is rather fragile, because the projecting ingress and exit tubes are apt to be broken off, with resulting fracture of the glass cylinder. Therefore, of far more practical use is the simple home-made condenser, consisting of a central glass tube passing through two corks, which effectually close the outer cylinder of uniform caliber. Through each of these corks passes an ordinary

piece of glass tube, which serves as an entrance and exit tube for the water (Fig. 72).

In this case the outer cylinder can be made either of glass or metal, and a very effective form of condenser can be made by using, as the outer cylinder, the ordinary tinned iron plaster box.

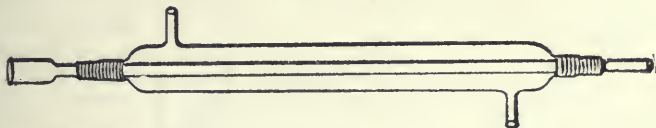


Fig. 71.—All-glass Liebig condenser.

Should the condensing tube become dirty, it can be washed with soap and water and scrubbed by passing through the tubes a plug of cotton attached to a stick or rod.

The only objection to the condenser just described is that the condensing tube, in order to be effective, must be of considerable length,

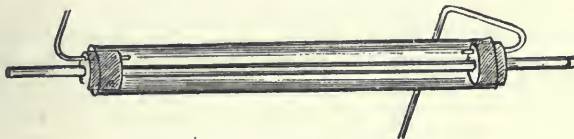


Fig. 72.—Corked Liebig condenser.

say three or four feet, and accordingly occupies considerable space when the apparatus is set up. In large processes of distillation, such as the manufacture of alcohol, a much longer condensing surface is required, and to make an effective straight condenser, taking, say, thirty feet, is hardly to be considered by reason of the space it would necessarily occupy.

This gave rise to a modification which answers the double requirement of occupying small space and of affording a large condensing surface.

The Condensing Worm.—This consists of a tube of glass or metal (usually block tin), twisted into a spiral and fitting into a vessel through which water can circulate freely. Such form of worm is used for all the large processes of distillation, such as obtaining alcohol and alcoholic liquors and distilling volatile oils. The objection to the worm, from the pharmaceutic standpoint, is due to the fact that its spiral form renders a thorough cleaning very difficult.

In technical operations a worm is used continually for the distillation of one substance, in which case a thorough cleaning is rarely needed, and this is accomplished by running water and other appropriate solvent through the tube. A combination of worm and Liebig condenser is shown in Fig. 73; it has value in a well-equipped organic laboratory, but is of little use in pharmacy because of difficulty of cleaning.

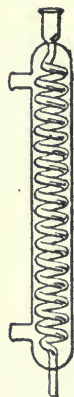


Fig. 73.—Spiral condenser.

Distilling Appliances.—In performing distillation with flask or retort and condenser, the fitting of the apparatus is a matter of considerable skill, and requires appliances not yet mentioned.

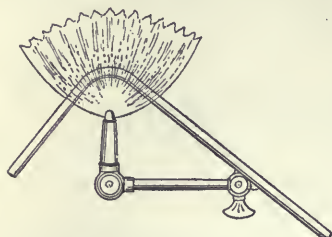


Fig. 74.—Bending tubing.

Glass tubing is usually needed to complete the connections, and a stock of good tubing should be at the disposal of every pharmacist, and with the manipulation of same he should be thoroughly acquainted. Such tubing comes in lengths of three or four feet, and can be easily broken into required lengths in the same way as glass rods, and, as in the case of glass rods, the edges should always be rounded by holding in a flame.

In bending glass tubing best results are obtained by first grasping one end, holding the tube over a fish-tail flame in such way that the end held by the hand is nearer the flame than is the other end. Rotating in the flame is not necessary, for, when sufficiently softened, the tube bends by its own gravity, and a round, elegant curve is obtained (Fig. 74).



Fig. 75.—Cork-borers (nested).



Fig. 76.—Rat-tail file.

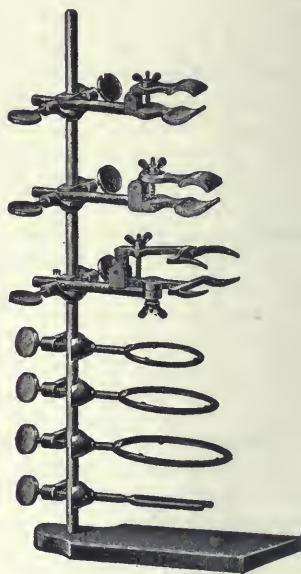


Fig. 77.—Retort stand.

The glass tube bent, the same must be inserted through the cork, which means that the holes must first be bored through the cork. This is done either by a rat-tail file (Fig. 76) or by a cork-borer, a set of

hollow brass tubes with lower end sharpened (Fig. 75), and the latter does the work so much more neatly that its use is strongly recommended.

To hold the condenser and the flask in place, we use clamps attached to a *retort stand*, a piece of apparatus of great value in all the operations described in this chapter, consisting, as it does, of support with clamps for condensers and rings for holding dishes during evaporation or percolators or funnels (Fig. 77). In the absence of such a stand the

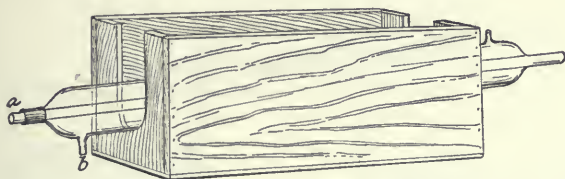


Fig. 78.—Box condenser holder.

ingenious and economic pharmacist can easily make an appropriate holder for his condenser from a wooden box, as shown in Fig. 78.

In connecting the condenser with the water mains, rubber tubing is essential, and very convenient for regulating the flow of water are the *pinch-cocks* which slip over the tubes and which can be opened or closed as shown in Fig. 79.

In making the connections between retort and condenser, rubber tubing is equally useful, although in processes requiring careful distillation contact of the vapor with rubber tubing should be avoided



Fig. 79.—Pinch-cocks.

as much as possible, in all cases it being advisable to have the glass tube from the retort introduced into the condensing tube (Fig. 80). The neck of most large retorts is usually larger than the opening of the condensing tube, and in such case, in order to avoid recourse to direct rubber connections, *adapters* are used, these consisting of tapering glass tubes which complete the "all-glass" connections between the retort and the condenser. For adapters nothing is better than the neck of a broken retort (Fig. 81).

In some cases of distillation care must be taken to prevent the pressure of the vapor from producing a fracture of the distilling apparatus,

or even explosions, and for such purpose safety tubes or funnels are used, as seen in Fig. 82. Those used in chemistry consist of a bent thistle funnel, in the elbow of which a few globules of mercury are placed, thus producing sufficient pressure to prevent a needless escape of the vapor, but being thrown up into the thistle bulb when the pressure within the flask becomes dangerous.

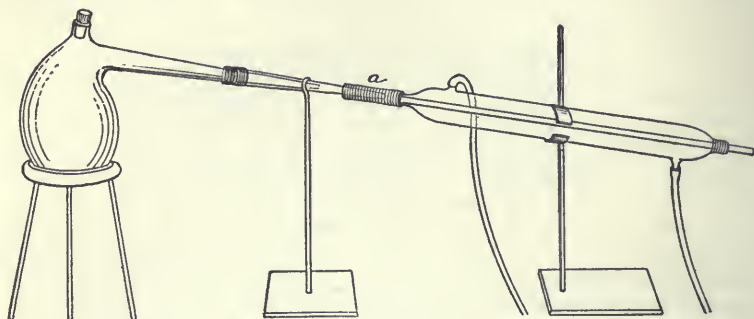


Fig. 80.—Fitting retort to condenser: a, Flaxseed lute.

Bumping.—This phenomenon is a disagreeable accompaniment of many cases of distillation occurring in a flask. In some cases the liquid does not boil steadily under application of heat, but at times the ebullition ceases for a few moments, then suddenly resumes, with a force sufficiently explosive to cause a spattering of the liquid, if not a fracture of the flask.

Bumping occurs chiefly with heavy and somewhat viscid liquids, which cause a retention of generated steam until a sufficient quantity has been formed to force its way through the opposing medium.

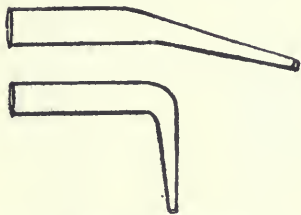


Fig. 81.—Adapters.

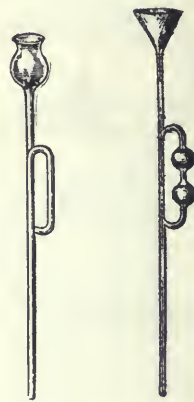


Fig. 82.—Safety tubes.

To eliminate this phenomenon, which very frequently spoils a tedious distillation by spattering some of the impure liquid into the condenser, and thence into the distillate, means to distribute the heat uniformly, and the insertion of pieces of pumice-stone, glass, or platinum has been suggested. None of these, however, answers as well as the maintaining of a minute current of steam or air through the distilling liquid.

Alembic still is that form in which the condenser is directly over

the boiler, and while the liquid distils more slowly in the alembic than in the retort form, it gives a pure distillate, as there is no danger of any of the liquid being carried over mechanically. A good commercial type of this form of still is the Phoenix condenser (Figs. 83 and 84).

Modifications of Distillation.—*Fractional distillation* is the method used for separating a mixture of several liquids of different boiling-points. Assuming, for example, we have a mixture of ether, boiling-point 37°C ., chloroform, boiling-point 60° to 61°C ., and alcohol, 78°C ., it seems easy to separate them by placing in a flask connected with a condenser and having a thermometer inserted in its neck, and gradually applying heat. When the temperature of 37° is attained, the ether should all distil over and be collected. Raising the temperature to

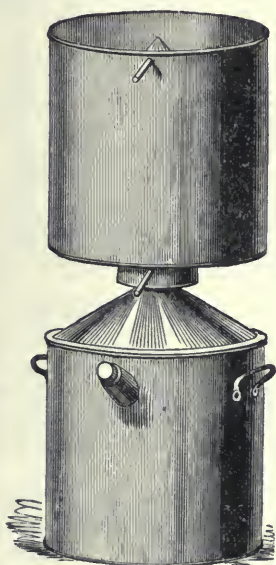


Fig. 83.—Phoenix condenser.

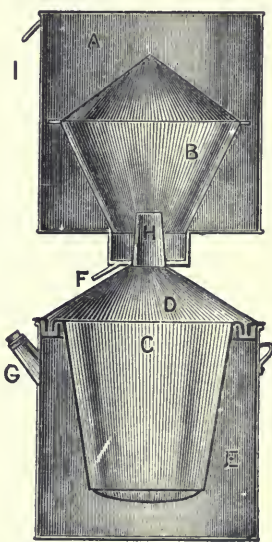


Fig. 84.—Phoenix condenser (outline).

A, Tank for cold water; B, condensing dome; C, boiler; D, connecting dome; E, water-bath; F, distilled water outlet; G, bath-water outlet; H, steam outlet; I, condensing water outlet.

60° to 61° , that portion should all distil over and be collected in a separate container, and, lastly, by raising the temperature to 78° , the residue, which should consist entirely of alcohol, should distil over and be collected. Unfortunately, however, the process is by no means as simple as this, for the reason that while it is true a liquid does not boil until a certain temperature is attained, the vaporization of that liquid occurs at temperatures other than its boiling-point, and, accordingly, the first distillate we obtain would be usually found to contain alcohol, chloroform, and ether, and it is equally true there is a strong possibility that the second and third distillates may contain some traces of ether, which had not been fully vaporized at the lower boiling-point.

Therefore, in order to carry out the distillation thoroughly, it is

necessary to take the three distillates we have obtained and redistill them, and experience has shown that a second repetition of the experiment will finally yield products which are practically chemical entities.

This process of repeated fractional distillation is largely used in chemistry, although scarcely at all in the ordinary pharmaceutical operations. By this means we are able to separate the volatile oils into their many constituents, and the same process is used for the separation and purification of the petroleum products.

Upright Condensation.—In some chemical and pharmaceutical processes it is found necessary to warm the substance for several hours with alcohol. To do this in free contact with air is an expensive undertaking, as alcohol evaporates very freely and would therefore pass into the atmosphere and be lost.

To permit economy in alcohol and similar valuable liquids, upright condensation has been devised. This consists (Fig. 85) of arranging the condenser in such relation to the flask that, as the vapors of the alcohol rise and are condensed, they are immediately dropped back into the flask, and by this means it is possible to keep alcohol at the boiling-point for weeks at a time without apparent loss.

A compact form of upright condenser is that devised by Soxhlet. This consists of two metallic hemispheres, the inner one communicating with the flask in which the liquid is heated, and the outer one with the water-supply; therefore, corresponding to the inner and outer tube of the Liebig condenser respectively (Fig. 86).

Some processes of distillation, as in the extraction of the volatile principles of drugs, are largely performed by intimately mixing the source of the volatile substance with water contained in the boiler, and heating both together, whereby the water and volatile constituents of the drug are volatilized and pass into the condenser, where they are liquefied and separated, the oil usually being immiscible with water.

Such, for example, is the method whereby oil of peppermint is obtained from the herb, and in all such cases of distillation care must be exercised to prevent the herb from coming in direct contact with the bottom of the vessel, the greater heat of which is apt to cause a partial charring of the organic drug, with subsequent decomposition of same, giving rise to the disagreeable tarry odor called "*empyreuma*." This decomposition is obviated by preventing the drug from resting on the bottom of the boiler, either by suspending the drug in an appropriate cloth, bag, or sack, or by having a still with a wire basket, such as is shown in the picture of the Remington still (p. 105).

Far better is the protection of the drug accomplished in the process

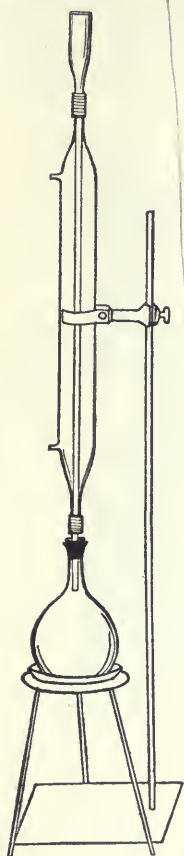


Fig. 85.—Invert condenser.

of *steam distillation*, which consists in generating the steam in an appropriate tin can provided with safety tube, and passing the steam thus generated into the flask or can containing the drug, and an appropriate quantity of water (Fig. 87).



Fig. 86.—Soxhlet's invert condenser.

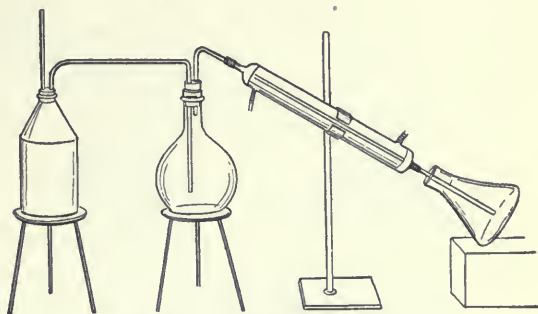


Fig. 87.—Steam distillation.

Through this the steam passes, carrying the volatile constituents of the herb into the condenser, where both become fluid.

Destructive Distillation.—In other pharmaceutic processes the decomposed products of the substances heated are desired. In some

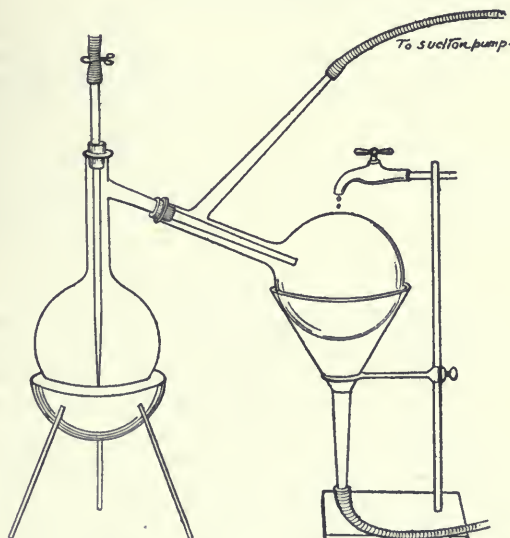


Fig. 88.—Vacuum distillation.

cases the substance is placed in a large iron retort and heated strongly, whereby the substance is decomposed and the volatile decomposition products pass over and are condensed by appropriate means. Such

condensation is used in the destructive distillation of wood, whereby we obtain the valuable commercial products of wood alcohol, acetone, and acetic acid. (See p. 645.)

Vacuum Distillation.—As mentioned on p. 92, evaporation is facilitated by removing air-pressure from vessel where the evaporation occurs, and at that place details for large operations of that character were given. In the chemical laboratory small operations of evaporation and condensation can be readily accomplished in the apparatus shown in Fig. 88, which, it will be seen, consists of two distilling flasks connected by air-tight rubber corks. One of the flasks acts as the boiler, while the other serves as condenser, and to the exit-tube of the latter is fitted the outer tubing connecting with the vacuum pump (p. 93).

In the neck of the flask acting as the boiler is fitted a rubber cork, through the orifice of which passes a glass tube drawn to a capillary point. The outer end of the tube is fitted with a piece of rubber tubing which is opened and closed by means of a pinch-cock. The object of this tube is to permit egress of sufficient air to prevent bumping; and if the temperature of the distillation is to be observed, the thermometer is inserted in the tube.

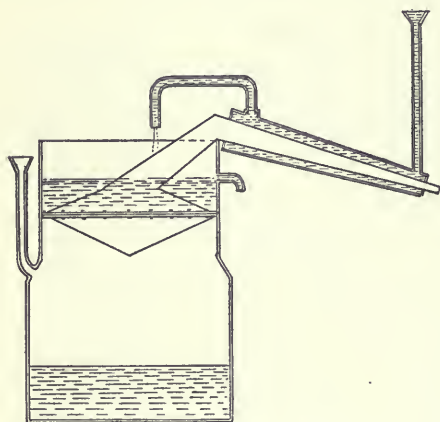


Fig. 89.—Curtman still.

The condenser is chilled by a stream of running water, while the boiler is heated by means of a Bunsen burner, with or without water-bath.

Special Stills.—As mentioned above, the word “still” is usually applied in the technic to a piece of apparatus, made of metal, consisting of boiler and condenser, and the varieties of these special stills are so numerous that space forbids more than mention of a few of the typical forms.

One of the most popular is the old-fashioned still with condensing worm, and, besides this, mention may be made of *Phœnix pharmaceutical condenser* (Fig. 84), which is of the alembic form; the *Curtman still* (Fig. 89), which is a combination of the alembic and condenser form, and the *Remington still*, in which the condenser occurs in a modified form of the Liebig condenser (Fig. 90).

A noteworthy feature in the Remington still is the arrangement of the condenser with seven parallel tubes placed in the same outer cylinder, on a principle akin to the tubular boiler. This modification gives a very extensive condensing surface in a comparatively compact form (as in a condensing worm), and possesses the advantage over the condensing worm that all tubes are straight, hence can be easily cleaned. This still is provided with a wire basket in which the drug desired to be distilled can be placed, thereby preventing contact with the bottom of the boiler and the consequent empyreuma.

The *Lloyd still* and concentrator works upon the principle that evaporation occurs rationally from the surface of a liquid, and that it is unnecessary to keep the contents of a still or an evaporator heated during the process. If the heat be applied to the surface of a liquid, the menstruum is instantly separated from the dissolved matters, which, dropping down into the still, are immediately cooled, and remain cool until the end of the operation. By automatically conducting a percolate to

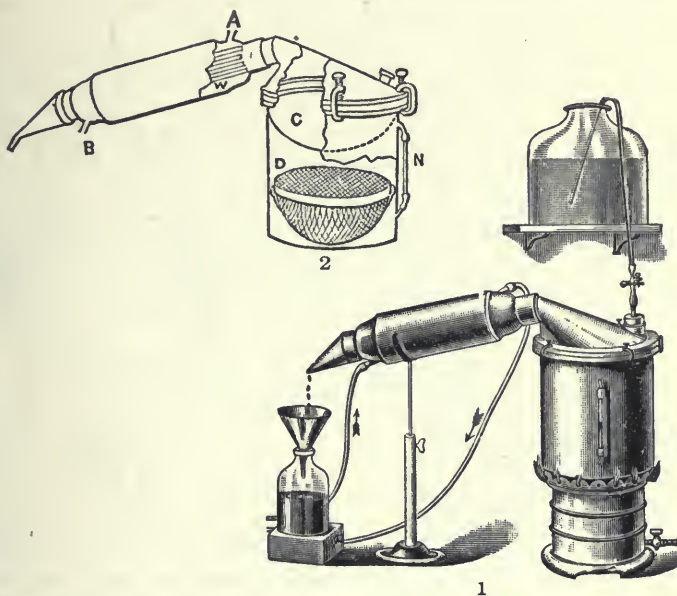


Fig. 90.—The Remington still: 1, Apparatus set up ready for use; 2, section showing condenser and wire basket. A, Condensing water inlet; B, condensing water outlet; C, boiler; D, wire basket; N, water gage; W, condensing tubes.

the heated surface, the distillate being conducted back upon the drug in the percolator, continuous distillation and percolation result. The application of the heat is instantaneous, whether the amount of drug worked be large or small. By this method the most delicate organic structures are preserved in solutions where a prolonged boiling would dissociate and destroy.

SUBLIMATION

This is the process of distilling a volatile solid, of converting the solid into a vapor, and condensing the vapor back to a solid.

A comparison of the three stages of the process shows how closely akin is the process to distillation:

Distillation.....	Liquid-gas-liquid.
Sublimation.....	Solid-gas-solid.

The object of sublimation, like distillation, is to obtain a volatile drug in a purer form. Sublimation is also one method of obtaining crystals.

As the apparatus employed in distillation was one of two types, so in sublimation the operation is carried on in apparatus of either the alembic or the retort type.

If the vapors are condensed directly over the vaporizing substance,

if the condenser is but little cooler than the temperature of vaporization, the sublimate will form in a solid mass—a *cake sublimate*. If, on the other hand, the condensing space is on one side, hence considerably cooler than the vessel in which the substance is vaporized, we get a fine powder—a *powdered sublimate*.

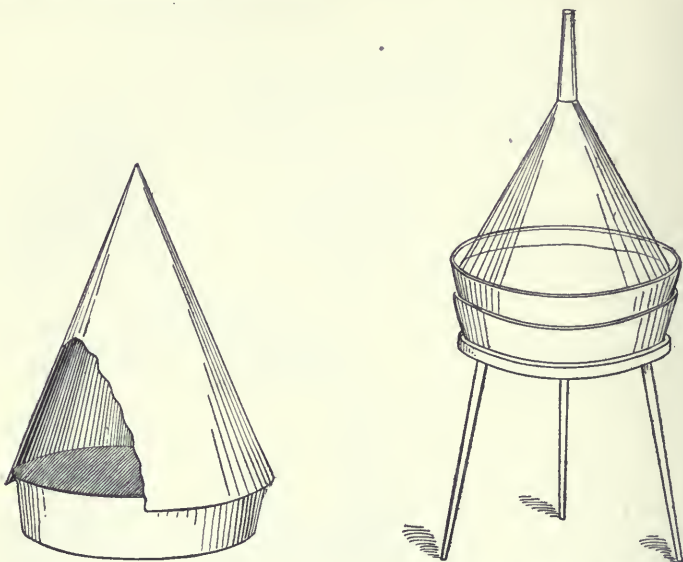


Fig. 91.—Subliming benzoic acid. Fig. 92.—Subliming camphor (small quantities).

No artificial means of refrigeration are needed in sublimation, the air being sufficiently cool to condense the vapor into a solid form.

Several of the official sublimed substances can spontaneously change from a solid to a gas, without assuming the intermediate form

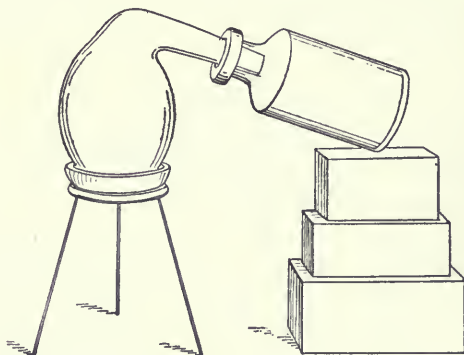


Fig. 93.—Sublimation of sulphur in glass retort.

of a liquid. This can be noticed in any bottle in which camphor has been kept, where handsome crystals, products of spontaneous sublimation, can be found in the upper part of the bottle.

Benzoic acid is another volatile official that sublimes to crystals. Not so, however, with the shining flakes of resublimed iodine, which are sometimes wrongly called crystals. Iodine is amorphous, as we will learn when we take up crystallization.

Apparatus.—Since in sublimation the condensation is usually effected at the temperature of the air, the apparatus used in the process is very simple, but similar to distillation in possessing the boiler part and the condensing part.

In small operations the alembic subliming apparatus consists of a dish covered with a cornucopia of paper, or with a glass funnel terminating with a wide-mouthed bottle (Figs. 91 and 92), while when powdered sublimates are desired, an ordinary retort is used as the boiler, and a wide-mouthed bottle as condenser (Fig. 93).

In the sublimation of large quantities of chemicals the apparatus is usually of cast iron, the condenser consisting either of the cast-iron dome over the retort, or a well-cooled room into which the neck of the retort projects. Apparatus of the first kind (Fig. 94) is used for the

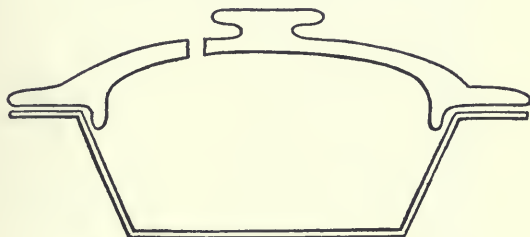


Fig. 94.—Subliming ammonium chloride.

sublimation of such cake sublimates as ammonium chloride; whereas the apparatus of the second kind is used for the sublimation of sulphur and the like, where a powder is desired.

DESICCATION

Desiccation is a *process of removing water from a substance at moderate temperature*, thus differing from exsiccation, which means removing the water from a substance at high temperatures.

Desiccation is one of the most commonly applied processes in domestic economy. For example, it would be perfectly correct to call the drying of a wet cloth by exposing it to air and sunlight “desiccation.”

Objects.—Desiccation is employed to aid in the preservation of the drug; to reduce the bulk of same; and to facilitate comminution.

That the drying of a substance aids its preservation by preventing the formation of mold is a fact well established, it being generally known that when most organic substances are allowed to remain in a permanently moist condition, moldiness and ultimate decomposition result.

It is a fact worthy of the pharmacist's attention, inasmuch as it is

a dictum that it is only a damp substance which becomes moldy, and, therefore, mold can be prevented by keeping the substance in a dry place.

That desiccation reduces the bulk of a substance can be shown in the drying of fresh herbs. Fresh herbs contain from 70 to 80 per cent. moisture, therefore the dry drug will weigh only about one-fifth of the fresh drug, and will occupy a much smaller space. The presence of moisture in a fresh drug renders it practically impossible to reduce same to powder, and if attempted by pounding the fresh drug in the mortar, a pulp results; hence it is given as a third object of desiccation that the process facilitates comminution.

Desiccation is not merely the drying of the fresh herbs, but means the removal of moisture from any drug; for example, if a chemical, like potassium acetate, absorbs moisture and becomes liquefied, there

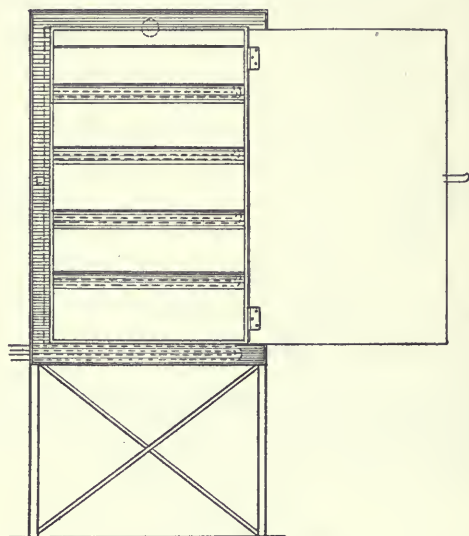


Fig. 95.—Drying closet.

is no need of throwing away the liquid mass, since by applying a gentle heat to the substance, the moisture will be eliminated and the dry chemical obtained.

In the same way, in making such pharmaceuticals as pills or lozenges, the freshly finished compound will be moist, and before putting same away, they should be freed from this moisture by desiccation. If such pills or lozenges are put away before completely dry, they will become moldy.

Apparatus used in desiccation is as varied as the different characteristics of the subjects to be desiccated. Thus in drying the herbs no apparatus is needed other than stout gunny bags, which are suspended in places having free access to air. In the smaller desiccations performed in laboratories several forms of apparatus are used. Among these mention may be made of *drying closets* (Fig. 95), which consist

of a cupboard, similar to a fume-closet, and, like it, having access to a flue, but arranged with a source of heat, such as a radiator, underneath. For still smaller operations *drying ovens* are used, these being heated by means of the Bunsen burner, and usually jacketed, so that a water-bath heat may be maintained if desired (Fig. 96).

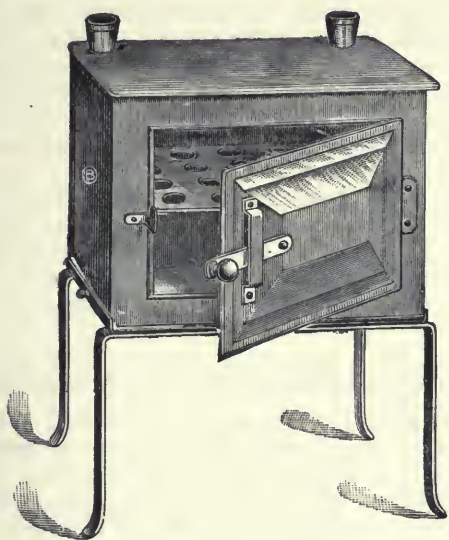


Fig. 96.—Drying oven.

To prevent a substance which has been desiccated from absorbing moisture from the air, recourse is had to *desiccators*, these usually consisting of a glass vessel provided with a ground-glass cover, the joints being made air-tight by tallow or petrolatum, the vessel being so arranged



Fig. 97.—Glass desiccator.



Fig. 98.—Hempel's desiccator.

as to contain in an appropriate space sulphuric acid or calcium chloride, both of which substances absorb water with great avidity, thus rendering the air within the container practically water free.

Of the two forms of desiccators illustrated in Figs. 97 and 98, the Hempel desiccator is arranged to permit a vacuum.

CHAPTER VI

COMMINUTION

THIS is the process of reducing a substance to finer particles, and, according to the fineness into which the particles are divided, we subdivide the subject into the branches cutting, slicing, chopping, rasping, grating, bruising, grinding, pulverizing, and triturating.

To obtain the substance in still finer form than that yielded by trituration, the processes of levigation and elutriation are used.

These several subjects will be considered in regular order, with mention of apparatus used in each case.

Objects.—The chief object of comminution is to present the drug in a form in which it is more susceptible to solution. In fact, with the exception of comminution for the purpose of making the so-called dusting-powders, all processes of comminution used in pharmacy are made with the intention of facilitating solution of the drug or its active principles.

VARIETIES OF COMMINUTION AND APPARATUS USED

Cutting, slicing, and chopping are familiar operations, rarely used in pharmacy, except for the preparation of drugs for the more complete processes of comminution. Cutting and slicing can, of course, be performed with any appropriate knife and chopping with any sharp instrument, such as a hatchet or cleaver, and in place of these familiar instruments, the best apparatus in pharmacy for the

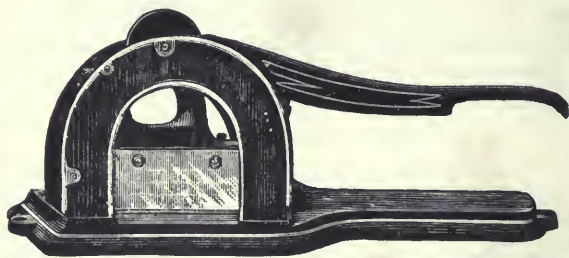


Fig. 99.—Herb-cutter.

purpose is the herb-cutter (Fig. 99). This can be used in cutting practically all drugs, and even for vanilla-beans, prior to making tincture therefrom. However, for the cutting of vanilla-beans, nothing is better than to take about a dozen beans, wind them tightly with a piece of stout twine, and slice them down with a sharp carving-knife.

Rasping and grating are not used very much in pharmacy, about the only drug which is convenient to rasp being guaiac wood, while grating is limited to the comminution of the small portions of nutmeg or lemon-peel, a nutmeg-grater being an inexpensive and convenient apparatus to have at a prescription counter.

Contusion or bruising is performed in a mortar, and for this special purpose the old-fashioned iron mortar is very useful. By means of

the instrument almost any drug can be reduced to powder of almost any fineness, but the process is so tedious that it has largely been replaced by grinding in a mill.

Other objections to the iron mortar are that pounding therein is a very noisy process, and a continuance of this pounding in a large mortar is apt to jar the building where the operation is conducted, hence such an apparatus should be used only on a firm foundation, and if on an upper floor, the mortar should be placed on a rafter and, if possible, over a supporting pillar.

Wooden mortars were formerly in considerable use, and at present are sometimes used in the bruising of acid drugs, such as beating up a tamarind pulp preparatory to making confection of senna, while huge marble mortars, which formerly enjoyed considerable popularity, have now almost completely fallen into disuse. Mortars used for contusion, the familiar form of which are seen in appended cut (Fig. 100), must not be confounded with the smaller mortars used in process of trituration at the counter.



Fig. 100.—Iron mortar.

Grinding and Pulverizing.—Grinding is the process of reducing substances to moderately coarse powders. Pulverization is a term usually applied to grinding to a still finer powder. In both cases the operation is carried on in special apparatus called mills.

A discussion of mills, as of stills, means mention of a vast variety of apparatus adapted for the various requirements of the trade, for example, in a thorough discussion of the subject, mention should be made of flour-mills and mills for other large technical processes. Even in the consideration of the strictly pharmaceutic operation of drug-grinding, almost as large a number of mills could be discussed, and picking from this array the most typical forms, it is necessary to discuss the following, but with a brief mention of their principle of action, leaving the cuts to convey the main characteristics.

Buhrstone Mill.—This is the ordinary form of mill, and is made of two stone corrugated plates which rotate on each other. On the rotating surface of the two are made grooves which act on each other, and on the drug interposed, in a manner somewhat similar to the blades of a scissors, clipping the drug to pieces at the same time that the weight of a stone crushed the drug to powder. Such form of mill is seen in the typical flour-mill (Fig. 101).

Roller-mill.—This consists of two large rollers of steel or iron, which revolve on each other and can, by means of suitable mechanism, be made to approach and become remote from each other. Such mill works entirely from the dead weight of the roller, and is used for the powdering of greasy or soft substances.

Chasers consist of two movable rollers rotating on a central axis on a steel base (Fig. 102). Such a chaser mill is used in the making of chocolate paste and the like.

Barrel or cannon-ball mills consist of barrels made of oak or steel, containing iron spheres about the size of cannon-balls, the barrel being arranged to rotate upon an axis, and when this is done, the cannon-balls roll to and fro with sufficient force to powder the drug contained

in the barrel. The advantage possessed by a barrel-mill is that the process of pulverization is made with but little loss, because none of the dust escapes. Its objection, however, is the intolerable noise of the rolling cannon-balls.

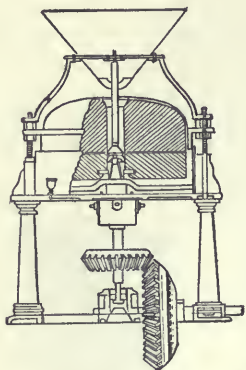


Fig. 101.—Buhrstone mill.

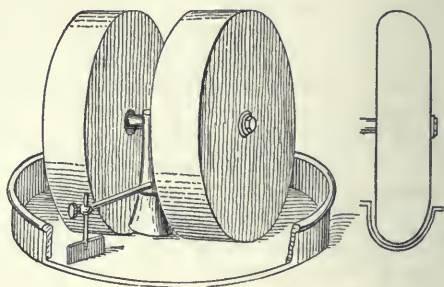


Fig. 102.—Chaser mill.

Mead's disintegrator is the most efficient type of the modern power drug-mill, and can best be understood by studying the cuts (Figs. 103 and 104). As seen therein, it consists of a steel disk having on the outer edge a series of steel projections. This disk rotates in a case the upper edges of which consists of a corrugated steel, against which the projections in the disk come almost into contact. The unique principle of the mill is the fact that it performs its work not by reason of its weight, but because of the enormous velocity at which the disk rotates, the drug placed in the mill being thrown up by the disk and its projections against the steel corrugations with a force somewhat akin to a cyclone and is so literally torn to shreds.

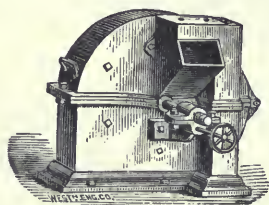


Fig. 103.—Mead disintegrator.

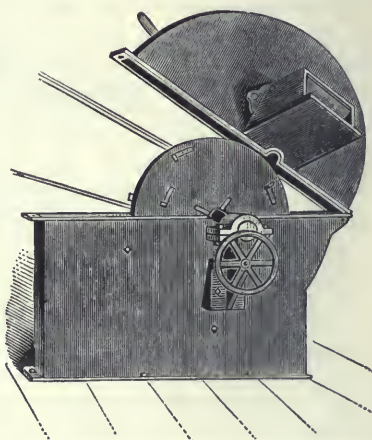


Fig. 104.—Mead disintegrator (open).

All the mills above mentioned operate entirely by steam power, and are, therefore, beyond the province of the retail pharmacy, hence of more importance to pharmacists are the mills now to be discussed.

Hand Power Mills.—Of the many varieties of hand mills, three types can be considered: Those in which the drug is crushed between

two vertical grinding surfaces, those in which the grinding surfaces are horizontal, and those in which the grinding surfaces are conic.

An example of the first type is found in the old-style Swift mill; the second type is shown in the Hance mill, and the third type is represented by the Enterprise mill, all of which are shown in the cuts (Figs. 105, 106, and 107).

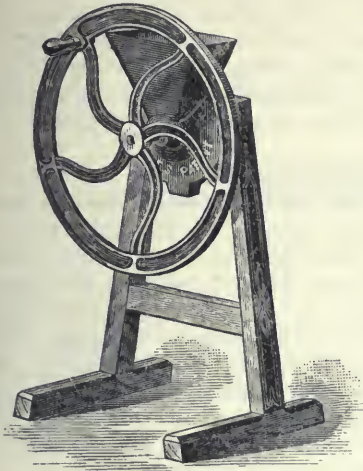


Fig. 105.—Swift mill.

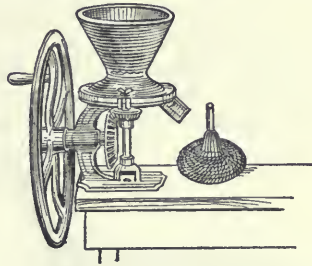


Fig. 106.—Hance mill.

Of these mills, the most serviceable is the conic variety, and in selecting a mill it is highly advisable to choose one in which the plates

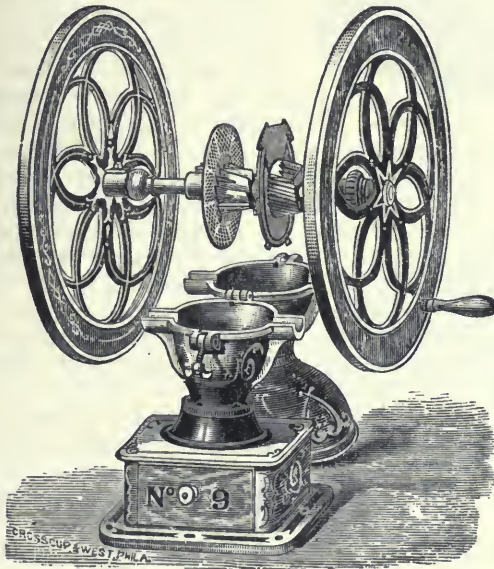


Fig. 107.—Enterprise drug-mill.

are easily removed for cleaning. By means of a drug mill, a substance can be rapidly reduced to powder provided suitable care is taken by

the operator, it being a good rule to first grind the substance coarsely, then return it to the mill, bringing the plates closer by means of the screws intended for that purpose, grind the drug again, and possibly repeat the operation with the screws still tighter. In rotating the plate by means of the crank or wheel, a uniform and steady motion should be maintained, far better results being obtained by a slow persistent motion than by rapid and spasmodic efforts. Under no circumstances should the mill be worked so rapidly as to overheat the plates.

With proper treatment a drug-mill should last a lifetime.

Trituration is the process of reducing a substance to a finer powder than is usually obtained by grinding in a mill, and is generally accomplished by placing a drug in a mortar and acting on same by the rotary action of the pestle. The apparatus used in trituration is the pestle and mortar aided by the spatula, and for trituration the pestle and mortar should be of the design shown in the appended cut (Fig. 108).



Fig. 108.—Triturating mortar.

Such mortars are constructed of porcelain, wedgewood, or glass, and for the trituration of a very hard substance agate mortars are sometimes used.

The pestle accompanying the regulation wedgewood mortar has usually a wooden handle which is put into the wedgewood base in a very crude way, usually by pouring a mixture of rosin and wax into the socket and placing the handle in while the mixture is still warm. Usually a pestle so fastened is very inconvenient, as the rosin and wax will chip

off and fall into the substance being trituated. If it is used in trituration of warm ointments, the wax is apt to soften, and if employed in mixtures containing alcohol, the alcohol is apt to dissolve the resin.

It is a good rule to fasten the pestle handle securely as soon as purchased, and this is easily done by removing the handle from the base by gentle warmth, cleaning the socket free from the resinous matter, and then pouring into the socket a thin mixture of plaster-of-Paris with water, insert the handle immediately, and allow it to stand until the mixture has completely set. A handle so fastened usually remains attached to the base.

As there is, however, some danger of the chipping of the plaster, a cement consisting of glycerin and oxide of lead (litharge) made into a paste has been recommended.

A wedgewood mortar after continual use becomes grimy and cannot be cleaned by the simple application of soap and water. To thoroughly cleanse such mortar, powder a few crystals of potassium dichromate in the same and rub to a thin paste with sufficient concentrated sulphuric acid; smear same over the mortar outside and in. When the paste is washed off, the wedgewood will be found perfectly clean.

Spatulas.—The object of the spatula in trituration is to scrape the triturated powder from the sides of the mortar, and for this purpose

a small spatula convenient to carry in the vest pocket is most convenient. Of the two forms of spatula shown in illustrations, the nickel-plated form is the most modern and most satisfactory, and every pharmacist should carry one as his personal property (Figs. 109 and 110).

Triturates.—Aside from the general term, trituration, as applied to reducing drugs to fine powder, a class of preparations called “trit-



Fig. 109.—Cocoa-handle spatula.

urates” have been introduced. These are of homeopathic origin and consist of the medicine intimately mixed with sugar of milk. The Pharmacopœia recognizes as this class of triturates preparations containing 10 per cent. of the active ingredient rubbed up with 90 per cent. sugar of milk. (See Triturate of Elaterin, p. 305.)



Fig. 110.—Nickel-plated spatula.

Sieves.—The grinding or compounding of a drug, no matter how carefully carried on, will not in itself produce an absolutely uniform powder, the more easily pulverulent portions of the drug being ground more finely than the denser portions. In order to insure a uniform powder, therefore, it is necessary to sift same through sieves, which consist of mesh work either of horsehair or metal stretched around a suitable holder (Fig. 111).

The sieve can be made with meshes of equal size, and for sifting drugs, sieves with twenty, thirty, forty, sixty, and eighty meshes to a linear inch are usually employed, the finished sieve being accordingly numbered, 20, 30, 40, 60, and 80 respectively.

In Germany and other countries of Europe the numbering of the sieve is different from the English and American, it being based on the number of meshes to the centimeter. As an inch is equal to 2.5 cm., the conversion of the German number to the American is not difficult, each unit of the German sieve being equal to 2.5 units of the American sieve. Thus a No. 15 German is equal to a No. 40 American (exactly $37\frac{1}{2}$); while a No. 20 German equals a No. 50 American.

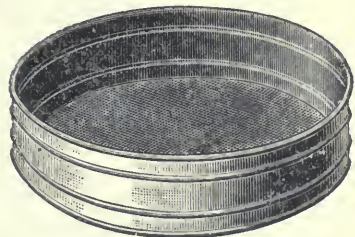


Fig. 111.—Brass sieve.

In using a sieve, the ground drug is thrown therein and the particles permitted to fall through the meshes by gently tapping the sieve with a spatula handle or the knuckle, particular emphasis being laid by

pharmaceutic writers against forcing the powder through the sieve by rubbing same with the hand. It is imperative that all portions of the same drug should be reduced to a uniform powder, hence we should not merely use the first portion that passes through, leaving the coarser particles for a subsequent grinding, but we should take a certain quantity of the drug, grind same with the mill, and sift, repeating the operation until the entire quantity has been passed through, except possibly a small amount of fibrous débris. When all the drug has thus been

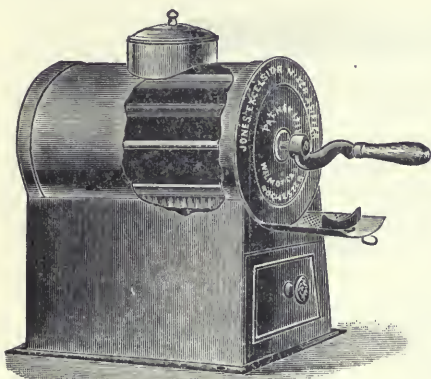


Fig. 112.—Excelsior mixer and sifter.

passed through the sieve, the powder is thoroughly mixed with the spatula, and only then can we say that we have completely powdered the entire drug.

Powdered drugs are given the same number as the sieve through which they pass; thus a No. 20 powder means a drug passed through a No. 20 sieve.

The finest sieve in ordinary use is the No. 80, and if the substance is desired to a powder finer than eighty, it is passed through bolting-cloth, which consists of a cloth with exact

meshes made of silk thread. The finer grades of flour are thus sifted through bolting-cloth.

In large drug-milling establishments the sifting is carried on in a power sieve, one of the best varieties of such an apparatus being the Excelsior mixer and sifter (Fig. 112).

Levigation is the process of reducing a powder to still finer particles than can be attained through trituration, and is accomplished by placing a substance on an appropriate slab, moistening with a few drops of alcohol, and smearing same over the slab by means of an

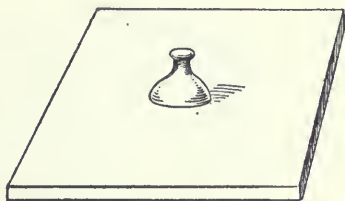


Fig. 113.—Slab and muller.

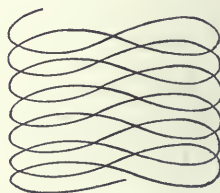


Fig. 114.—Movement of muller.

instrument known as the muller (Figs. 113 and 114). As shown in cut, the correct way of using the muller is by rotating the same in a circular motion, as if drawing the figure 8. If the slab and muller are composed of porphyry, the process is called *porphyrisation*.

Elutriation is a crude process of separating coarser from finer particles by suspension in water, hence is sometimes called "water-sifting." It is used only in separating insoluble substances, and is

limited to a few cases, such as the separation of chalk from the grit with which it is associated when mined. The freshly mined chalk is coarsely ground, mixed with water to a thin paste, and then thrown into a tank containing water, with the result that the gritty quartz will be washed out and subside, while the less heavy chalk remains for a time suspended in the liquid. After a few minutes the liquid is drawn from the tank into another through a faucet, placed some distance above the bottom of the vessel, and there is allowed to stand until the suspended chalk has separated, whereupon the clear liquid is drawn off and the pasty chalk is obtained almost free from grit. In commerce this chalk paste is usually marketed in the form of cones, and these are produced by the process known as trochiscation.

Trochiscation.—By placing the paste in a wooden vessel shaped like a funnel, and by giving the apparatus sharp raps, the paste drops upon a porous surface, such as a slab of chalk or of earthenware, where it dries in cones, an expert workman being able to make practically all the cones the same size.

Pulverization by Intervention.—There are some substances which are almost impossible to reduce to powder by trituration or other methods of comminution already discussed. Among these the best known example is camphor, which, when pounded with a pestle, becomes a pliable mass. If, however, to the camphor a few drops of alcohol or chloroform be added, it can be powdered without difficulty. This method of reducing a substance to powder by means of another substance is called pulverization by intervention. Camphor powdered with alcohol is apt to cake on standing, and this can be obviated by adding to same a minute quantity of colorless petrolatum.

Other examples of pulverization by intervention are the powdering of gold-foil by trituration with potassium sulphate, the potassium sulphate being removed after trituration by treatment of the powder with water. Tin can be powdered by pouring the molten metal into prepared chalk and shaking until thoroughly cold, the prepared chalk being separated from the powdered tin by treatment with diluted acids. Phosphorus can be powdered by simply melting it in solution of salt and shaking same until cold.

CHAPTER VII

SOLUTION, LOTION, DECANTATION, COLATION, AND FILTRATION

SOLUTION

THE time-honored definition of solution, that is, a process whereby a solid or gas is liquefied by means of a solvent, is scarcely the modern view of the subject.

The best definition is a "molecular subdivision of a body in a liquid, the result being a clear, homogeneous fluid." By "molecular

subdivision" is intimated that all substances going into solution—be they solids, liquids, or gases—become divided from masses into *ions* which permeate the molecules of the dissolving liquid, producing homogeneity or uniformity.

Thus each molecule of sodium sulphate, on being dissolved in water, is separated into positive ions consisting of Na, into negative ions, consisting of SO_4 , a molecule of water acting as the partition between each of the ions.

Solvent.—In order to dissolve a substance a solvent is required, and the character of different solvents varies greatly, investigation showing that practically all physical combinations, such as the making of solders, fusing of alloys, and the like, follow the same rules as does the ordinary solution. Thus, at present, alloys, such, for example, as brass, are considered a solution of the metals of which it is composed.

Pharmaceutically, however, by a solvent is meant a liquid in which the substance is made to disappear and lose its identity.

Of the solvents used in pharmacy, *water* is preëminently the most valuable, as in it dissolve most inorganic chemicals, and such organics as sugars, gums, and glucosides.

Alcohol dissolves resinous drugs, volatile oils, etc.

Glycerin is an excellent solvent, dissolving the water-soluble substances, and in larger quantities, but its stickiness precludes its general use.

Acids are used as solvents in some cases, although usually the acid effects a chemical change in the nature of the dissolved substance.

Alkalis are valuable solvents for some substances, as albuminous matter, waxes, starches, and resins, but they, like the acids, usually make chemical changes in the dissolved substances.

Chloroform and *ether* and *petroleum ether* (or *benzin*, U. S. P.) are solvents for resins, oils, and fats, though their use is somewhat limited.

Carbon disulphide is used for dissolving rubber, while for solution of gutta-percha, chloroform answers.

Of late, in chemical work, acetic ether has become a valuable solvent, while *wood-alcohol* and also *acetone* are largely supplanting grain-alcohol in many cases of solution in the arts.

Solubility.—By this term is meant how much of a substance dissolves in a given solvent. The range of solubility is practically limitless, some substances in much less than their own weight of the solvent, whereas other substances require many times the weight of the solvent. Thus, silver bromide requires one to two million times its weight of water for solution, while potassium iodide dissolves in seven-tenths its own weight of water. When a substance is so slightly soluble as silver bromide, we usually term it insoluble, although practically every substance does dissolve in minute amounts, as in the case of the chemical just mentioned.

To memorize the individual solubilities of all the officials is practically impossible, although the student should know in a general way whether a body dissolves in water or is insoluble, and, moreover, he should be acquainted with the approximate water solubilities of such well-known chemicals as potassium bromide, potassium iodide, and potassium chlorate.

Regarding general solubilities in water, the following information

relative to inorganic salts is useful, especially in considering prescriptions:

SOLUBLE.

All acetates.
All nitrates.
Sulphates, except barium, strontium, lead, silver.
All sodium salts.
Potassium salts, except bitartrate.
Chlorides, except silver and mercurous.

INSOLUBLE.

Carbonates, except those of the alkalis.
Phosphates, except those of the alkalis.

This table is of barest outline, and refers only to well-known medical substances. For instance, there are a few insoluble sodium salts, but they are not worth consideration in pharmacy. Then remember that the term "soluble" is purely relative. Potassium iodide is soluble in three-fourths its weight of water at 15°C .; calcium sulphate, on the other hand, requires 410 parts of water at 15°C . to dissolve it; yet both are classed as "soluble" in above table. Then in the table many substances are missing. For instance, no specific mention is made of copper and zinc salts, or of the silicates, or of a host of other bodies. These omissions are due to the fact that no classification of the substances as to the solubility can be made. Some zinc salts are soluble, some are insoluble; hence they are omitted.

Simple and Chemical Solutions.—Thanks to the investigations of Arrhenius and Ostwald, the former popular distinction between simple and chemical solutions has become almost obsolete, Arrhenius showing that when the chemical dissolves in water, it immediately dissociates into two or more portions, called "ions," and, in fact, in exactly the same way as some substances separate when under the influence of electricity. In the old method of reasoning a solution of Glauber salt, Na_2SO_4 , in water was a simple solution, but if the same salt solution was produced by mixing a solution of sodium hydroxide with dilute sulphuric acid, because a chemical action occurred during the process of solution, this latter solution of sodium sulphate was a chemical solution.

The modern view is that both solutions are identical, consisting as they do of ions of sodium and ions of the SO_4 group; hence in the modern discussion of solution the terms "simple" and "chemical" are eliminated.

However, in the pharmaceutic discussion of the solutions of the United States Pharmacopœia it is convenient to classify (same) into those made by simply dissolving the solution in water, which solutions are called "galenics," and those in the manufacture of which by the pharmacist a chemical change occurs. In this sense the term simple and chemical solutions are still applied, without, however, any intention to intimate that any marked difference exists between the same compound made by simple solution and that made by chemical action.

Conditions of Solution.—In order to facilitate solution it is advisable to pulverize the substance which is to be dissolved, the *advantages of pulverization* being best illustrated by a homely simile.

Imagine a closed book of 500 pages as a lump or crystal of matter. Imagine this exposed to the action of a solvent. This solvent can only attack front, back, and four edges—a surface scarcely equal to that of three leaves. Now imagine that book taken all apart, and its 500 pages spread flat on the floor, and see what an enormous increase in surface. In the same way a great increase is produced in the surface exposed to the action of a solvent when a crystal is reduced to powder.

After powdering the substance and mixing it with the solvent, solution can be facilitated by *agitating* the mixture of the substance and the solvent, and this, because, when we suggest that a solution is a minute subdivision of the substance affecting the very molecules which permeate the molecules of the solvent, it is plain to be seen that in order completely to mix these molecules, agitation is necessary just as some form of agitation aids in the mixing of solids.

Among the conditions of solution the *character of the solvent* has considerable influence, and it is, of course, self-evident that it is useless to attempt a solution of a substance in a solvent which does not effect it. In the same way care must be had in mixing solutions not to add to the liquid another liquid which is apt to precipitate the dissolved substance, even though that liquid is freely miscible with the solvent that has been employed. Thus, alcohol and water dissolve in each other, but if in the water is dissolved a substance which will not dissolve in alcohol, such, for example, as gum arabic, the addition of more than traces of alcohol will result in the precipitation of the gum from the solution.

In the same way, if we have an alcoholic solution of a resinous substance and add water to it, the water will mix with the alcohol, but in the process of solution of the two fluids the resin, which is insoluble in water, is precipitated.

Temperature is a decided factor in influencing solution. Usually a high degree of heat facilitates solution, although not in a definite ratio. Thus one part of potassium chlorate will dissolve in 16.7 parts of water at 15° C., while one part of the same salt will dissolve in 1.7 parts water at 100° C. This is not always true, however, some substances being more soluble in cold water than in hot.

Thus one part of lime will dissolve in 750 parts water at 15° C., while it takes 1300 parts of water at 100° C. to dissolve the same quantity.

An interesting case of temperature variation in solution is found in sodium sulphate (p. 475).

Heat aids solution in rendering molecular subdivisions easier. A body, in fusing, changes from a solid to a liquid, simply because the heat changes the condition of the molecules from fixed rigidity to the mobility characteristic of liquids. This is supposed to be caused by the influence of heat in lessening the cohesive force of the molecules of the substance—by pushing the molecules farther apart. In the case of solution, the molecules of dissolving substance becoming separated by diffusion among the molecules of the solvent, the separation—the pushing apart of the molecules—is greatly facilitated by the application of heat, and that its aid in overcoming cohesion is usually enlisted in solution is shown by the fact that, when a substance is dissolved at normal temperatures, it usually takes up so much heat away from the solvent that the liquid becomes cold. This is especially to be noticed dissolving potassium iodide in water, and freezing mixtures are usually devised on this principle. However, bear in mind that all solids do not chill the solvent on dissolving. For instance, when calcium chloride is dissolved in water, the temperature of the solution is raised considerably. This, however, is supposed to be due to a chemical combination with water, as is also assumed to be the case when sulphuric acid or alcohol is mixed with water.

Method of Solution.—The ordinary method of dissolving a substance is, as suggested just above, by reducing the substance to a comparatively fine powder in a mortar, then adding the solvent, and triturating until solution is accomplished, and finally filtering the

finished solution by passing through a pledget of cotton placed in an appropriately sized funnel. The careful pharmacist, in preparing solutions on prescriptions, should always observe this rule, as the chemicals used are likely to contain minute traces of insoluble impurities, such as fragments of cork from the container, or possibly traces of soot, and to dispense a solution any way other than absolutely clear does not enhance a pharmacist's reputation. In a case of a very soluble salt, such as potassium iodide, the powdering and subsequent trituration in the mortar are not necessary, in such case the solution being quickly effected by throwing the crystals into the flask designed to hold the finished prescription, adding the water, shaking until solution results. In all such cases the rule of filtration through cotton should be rigidly adhered to, it being a simple matter to pour the liquid from the flask into a graduate and then filter into the bottle.

Some substances, such as gum arabic and sugar, dissolve comparatively slowly, and in such cases recourse is had to that method of solution known as circulatory solution.

Circulatory Solution.—In this process the substance is tied in a piece of cloth and suspended just below the surface of the solvent. The liquid in contact with the drug becomes impregnated with the substance, and, being of greater density than the water, sinks to the bottom of the container, giving place to a fresh supply of water, which in turn becomes denser and sinks. In this way a constant downward current of dissolved substance and an upward stream of displaced water produces a circulatory motion of the liquid in the container, hence the name, "circulatory solution."

Saturated solution is one which contains all of a certain substance that the solvent will hold in a solution, and the quantity of a drug in such a saturated solution is, of course, dependent on the relative solubility of the drug. Thus, a solution of lime containing in 100 grammes only 16 centigrammes of calcium hydroxide is a saturated solution, even as is a solution of potassium iodide, 100 grammes of which contain about 56 grammes of potassium iodide. In other words, the term saturated means the limit of solubility rather than the comparative denseness of solution. An interesting point connected with saturated solution is that a saturated solution of one substance may act as an admirable solvent of another; in other words, in a saturated solution, the solvent powers of that liquid are exhausted only as far as that one substance is concerned, but can be exerted in effecting solution of another substance. Thus a saturated solution of potassium iodide not merely dissolves iodine, but also much more of that chemical than will water alone. In the same way a saturated solution of potassium nitrate will freely dissolve copper sulphate, and this scheme has been used for removing copper impurities from large quantities of potassium nitrate.

Supersaturated Solution.—Under certain conditions, a solvent sometimes takes up and holds more of a salt than is indicated by the solubility of that salt.

This is particularly the case when the salt is treated with water of a temperature greater than normal. As already mentioned, hot water will usually dissolve more of a salt than will cold water, and such solution, on standing, usually deposits the amount of salt in excess of

the quantity soluble at ordinary temperatures of the atmosphere, and, indeed, as will be noticed in crystallization, this great excess usually draws with it a great majority of the salt in solution.

A solution of this kind, holding more of the salt than is normally dissolved, is said to be a supersaturated solution, and in some cases such a supersaturated solution will remain liquid without depositing the excess until violently agitated. Such is particularly striking, as shown in the case of sodium sulphate (p. 475).

Volume Changes in Solution.—In effecting solution between a certain volume of a chemical and a certain volume of its solvent, the resulting solution usually occupies less space than do the two ingredients. In other words, contraction of volume occurs in effecting solution. By this, of course, reference is not made to the fact of the solvent occupying the interstices or air-spaces found in a certain volume of a crude chemical, but what is meant is the actual volume of the solvent itself independent of air-spaces. Thus sugar (specific gravity, 1.336) has a specific volume of 0.748, and 85 Gm. of sugar, therefore, would occupy the space of 63.6 Cc. This 85 Gm. of sugar, dissolved in 48 Cc. of water, made 100 Cc. of syrup, showing a contraction of 11.6 Cc. in the process. In the same way dried alum dissolved in water contracts, as does the well-known mixture of alcohol and water in preparing diluted alcohol.

The latter case of the contraction of alcohol and water in preparing diluted alcohol is sufficiently marked to be worthy of attention by the operator, as was shown by an incident coming to the author's attention, in which a pharmacist, in preparing forty gallons of dilute alcohol for a customer, mixed twenty gallons of alcohol and twenty gallons of water, bottled same without measuring, and was much grieved on receiving complaint from the customer that the quantity was several quarts short.

Estimation of Solubility.—Reference has been made in a preceding paragraph to the solubility of a solid in water and other solvents. This is done in several ways, the usual method being by weighing a certain quantity of the solid in a tared bottle, adding a certain quantity of water at a given temperature, usually 15° C., shaking until assured of a saturated solution, then carefully transferring the liquid to a quick-acting tared filter and then weighing the solid residue. In this way we learn the amount of the solid which has been dissolved, and the solubility of the solid can be easily estimated on the basis of the solubility of one gramme of the solid; thus, if the experiment shows that one gramme of salt dissolves in three grammes of water, then the solubility is put down as one part in three of water. This method, however, is not exactly accurate, inasmuch as during filtration a certain amount of the solvent volatilizes, therefore leaving a greater residue than that actually soluble. A more accurate way is to take a given quantity of the solution, evaporate same to dryness, and from this estimate the solubility of the solid. In the latter case, however, the contraction of a solid in the solvent must be known, and this factor used in the estimation of the solubility. For simple pharmaceutical purposes a rough estimate of the solubility can be easily obtained by adding the substance to the liquid until no more is taken up. Thus if to 100 Cc. of water at normal temperature a solid is added in 5-gramme

lots, the solubility of the salt can be estimated within 5 per cent., and to attain absolutely accurate results it is only necessary to repeat the experiment by dissolving in the water the amount of salt within 5 grammes of supersaturation, and then add the rest of the salt in half-gramme lots until no more is taken up.

For very accurate estimation of solubility recourse is had to the lysimeter of Dr. Rice, while an absolutely accurate method of the solubility of alkaloids by use of volumetric solutions has been suggested by Hatcher (*"Amer. Jour. Pharm.,"* 1902, p. 134). Attention has been called to the fact that in order to avoid supersaturation in estimating solubility the mixture of the solid and its solvent should be thoroughly agitated by means of a mechanical stirrer during the process of solution.

Gaseous Solution.—While physically a solution of a gas in a liquid differs but slightly from the solution of either a solid or a liquid, the mode of preparing a gaseous solution differs sufficiently from the

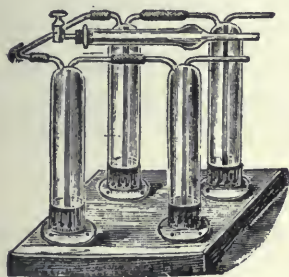


Fig. 115.—Apparatus for drying gases.

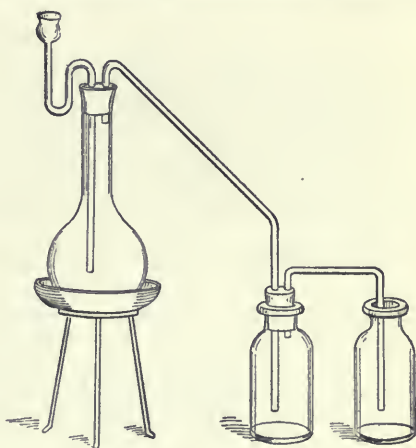


Fig. 116.—Gaseous solution.

solution of either a solid or a liquid to justify a special heading. In effecting the solution of a gas in water when the gas is freshly generated or supplied from a storage tank (in which form liquid carbon dioxide is used largely for charging soda fountains), it is first washed by passing through water and then conducting it through a tube reaching almost to the bottom of the container holding the liquid, and to insure solution it is best that the container be long and narrow. When the soluble gas is run into the liquid, it is noted at first that the large bubbles emerging from the end of the tube are absorbed before they reach the top of the liquid; as the solution becomes more and more saturated it is found that the bubbles are less dissolved, until finally it can be noted that the size of the bubbles on emerging from the top of the liquid is about the same as when emerging from the end of the tube. When this stage is reached, it is always advisable to remove the container with the liquid and shake vigorously, again pass gas through, and if it is found by this means that no more gas is being dissolved, the operation is complete.

A simple test of saturation of a liquid with a gas is by observing the behavior of the solution after agitation. Place the hand over the mouth of the container, and if, after each agitation and attempted removal of the hand from the mouth of the bottle, a suction is felt, it is a strong evidence that the solution is not saturated. If, on removing the hand, a pressure of the escaping gas is observed, it is rather a fair indication of complete saturation.

In some cases, before making a solution of the gas, it is essential that same be dried, that is, freed from water vapor, in which case, instead of running such gas through water, it is passed through concentrated sulphuric acid, calcium chloride, or other substance having great affinity for water, contained in appropriate flasks (Fig. 115). There are some gases which cannot be dried with calcium chloride nor sulphuric acid, such, for example, as ammonia gas, and in such cases the gas can be fairly well dried by passing it through a bottle containing absorbent cotton. An illustration showing gaseous solution is appended (Fig. 116).

LOTION

Lotion is derived from the Latin "lotio" (to wash), and means the removal of soluble impurities from insoluble substances by washing same with water or other appropriate liquid. Thus, in the last stage of purifying precipitated calcium carbonate, the precipitate contained on the filter is washed with water, while in making washed sulphur the sublimed sulphur is washed with a mixture of water and ammonia water.

Apparatus used in lotion consists of funnel with a filter-paper, or straining rack containing an appropriate strainer. (See Colation.) On this filter or strainer is thrown the substance mixed with the wash liquid, the latter, of course, passing through the strainer, the meshes of which hold back the insoluble matter. Upon the precipi-

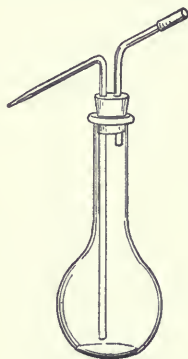


Fig. 117.—Spritz bottle.

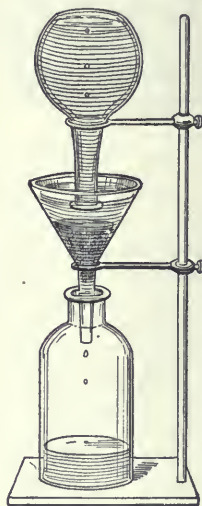


Fig. 118.—Continuous washing.

tate contained on the strainer, water or other wash liquid is poured until all of the soluble impurities have been removed, as can be ascertained by appropriate tests.

When only a small quantity of washing is required, the liquid is squirted upon the filter from a Spritz bottle, which consists of an ordinary pint chemical flask provided with a two-holed rubber stopper, through one hole of which passes a glass tube extending almost to the bottom of the flask, and above the cork bent at an acute angle and with the outside end drawn to a tapering point. Through the other hole is passed a shorter piece of glass tubing bent at an angle of 120–140 degrees. The water is removed from the Spritz bottle by blowing through the shorter piece of glass tube, when the increased pressure of air inside the flask will cause the liquid to pass through the longer glass tube, and emerge from the tapering end in a stream. If this stream be directed to the precipitate on the filter, the latter can be thoroughly and easily washed (Fig. 117).

Where large quantities of a liquid are needed for washing, it is a simple matter to devise a continuous process of washing, as shown in appended cut (Fig. 118), in which it will be seen that a flask filled with water is inverted into the funnel containing the precipitate on the filter, upon which the liquid rushes until it reaches the mouth of the flask, when the flow ceases, owing to the inability of the air to pass into the flask. As the liquid of the funnel filters, its surface becomes lower than the mouth of the flask, thus permitting the entrance of air, causing a flow of the washed liquid from the flask until its mouth is again closed by the liquid in the funnel, and this continues until the flask is emptied, and that without danger of the liquid running over the edge of the funnel. Such simple apparatus is of great value in continuous filtration, in which case it is a simple matter to filter gallons of a liquid through a half-gallon funnel.

DECANTATION

In some cases it is more convenient to wash a precipitate by placing it in an appropriate glass vessel, such as a beaker or a flask, mixing thoroughly with a wash liquid, and allowing it to stand until the precipitate has completely subsided, and then removing the liquid by decantation; that is, by gently lifting the container and gradually tilting same until the major part of the liquid has been removed from the precipitate. It is manifestly impossible by this means to thoroughly remove all the liquid, as no matter how carefully decantation is continued, certain portions of the precipitate will be disturbed and flow out with the decanted liquid which, therefore, should be passed through a filter to collect the escaping precipitate. Repeated decantation, however, will enable us to thoroughly remove soluble matter from the precipitate. Thus, a precipitate contains 100 grammes soluble impurities: by treating with 1000 Cc. water, the 100 grammes soluble matter will dissolve. By decanting this, it is possible to remove 900 Cc. of the liquid without seriously disturbing the precipitate, and in 900 Cc. 90 Gm. of soluble impurities will be contained. To the remaining liquid and the precipitate another 1000 Cc. water is added, the product thoroughly mixed, and the precipitate allowed to settle. When 900 Cc. of this second liquid are removed, it means that of the 10 Gm. soluble substance remaining, 9 Gm. will have been removed, leaving with the precipitate but one gramme of the original 100. The operation repeated,

the 900 Cc. of the decanted liquid will remove 0.9 of the remaining gramme of the soluble impurity, and thus, after three such washings, but one one-thousandth of the original impurity remains.

Ordinarily, however, such thorough washing by decantation is not carried on, as it means the use of too much water.

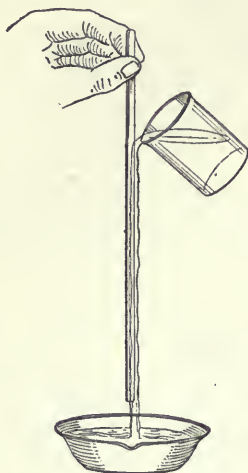


Fig. 119.—Decantation from beaker.

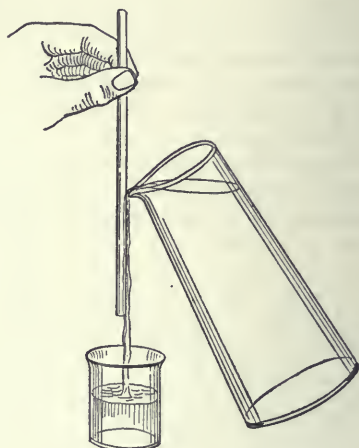


Fig. 120.—Decantation from precipitating jar.

Apparatus for decantation is the container mentioned above, consisting either of a pint flask or a beaker, provided with a pour-out or lip.

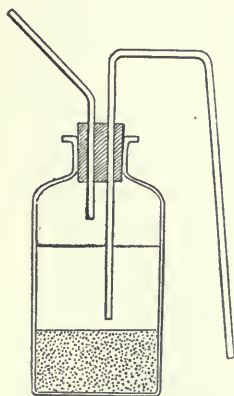


Fig. 121.—Syphoning.

To facilitate the removal of the decanted liquid, a glass guiding rod is used, such as was described on p. 91. The method of application is best shown by appended cuts (Figs. 119 and 120). To avoid the possibility of any of the liquid being drawn over to the outside of the beaker by the capillarity of the glass, the edge of the beaker should be smeared with traces of some fat. The guiding rod is of great value in quantitative analysis, in which case every particle of the precipitate must be finally removed from the beaker and weighed. This is accomplished by slipping a small piece of rubber tubing over the end of the guiding rod and rubbing the sides of the beaker with same. Such rubber-tipped guiding rod is technically called a "policeman."

Where large quantities of liquid are to be decanted, recourse is had to the **syphon**, which consists of a bent tube, one arm of which is longer than the other. The shorter arm is placed in the liquid to be removed, and at the other end of the tube suction is applied (Fig. 121).

We take a syphon, immerse the short arm in water, and suck at the orifice of the longer arm. This has the effect of producing a partial vacuum in the tube—a pressure of air therein much less than the pressure of the atmosphere exerted upon the surface of the liquid in which the short arm is plunged. The result is that the superior pressure of the atmosphere forces the water into the syphon through the short arm, around the bend, into the long arm—this result, of course, being simultaneous with the suction applied at the end of the long arm. Entering the long arm, the liquid is subjected to the earth's gravitating force and falls downward. As each drop emerges from the syphon, were nothing to take the place occupied by that drop, a partial vacuum in the syphon tube would occur. Equilibrium—an exact balance between two parts of a machine or system—is the rule of nature, hence difference in pressure in the long and short arms of the syphon is not permitted, and the instant a partial vacuum is produced in the long arm, the water in the short arm and in the vessel into which the syphon dips, impelled by an atmospheric pressure, passes over to occupy the void.

Just as rapidly as water emerges from the long arm, even so rapidly does water pass over from the short arm to take its place, and this condition continues so long as the short arm dips in water and the level of this water is higher than the level of the water that issues from the long arm.

COLATION

Colation is the technical name for the simple process of straining, by which term is meant the removal of coarse particles from a liquid by passing through a fabric the meshes of which are comparatively coarse. Thus, the domestic utensil, the colander, is useful in colation. In the same way a sieve can be used for the purpose of straining a liquid, but ordinarily the fabric through which the substance is passed is of some form of cloth. Among the various cloths used for straining are felt, flannel, muslin, and cheese-cloth.

Felt strainers (Fig. 122) are usually of conic form and yield a liquid as clear as if filtered. Equally useful is a strainer made of flannel (Fig. 123), and these are generally sewed together in the same conic form as the felt strainer, while the muslin and cheese-cloth strainers are simply pieces of these fabrics fastened to a frame (Fig. 124). The latter consists of four pieces of wood, joined and studded with projecting nails, on which can be fastened the straining cloth. The latter should fit tightly to the frame without edges projecting, as in the latter case each filament of the cloth acts as a syphon and part of the liquid runs over the sides of the strainer to the container in a way that scarcely betokens pharmaceutical elegance.

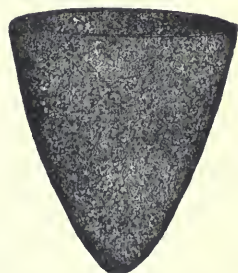


Fig. 122.—Felt strainer.

In many cases of straining the operation has been aided by what has been termed "forcible straining," which means removing the straining cloth from the frame, gathering between the hands by a twisting movement the strainer and the residue thereon, and then

expressing (p. 153). Straining is an imperfect operation, inasmuch as the strained liquid is rarely as clear as the careful pharmacist desires

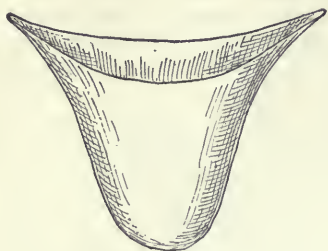


Fig. 123.—Leg-of-mutton strainer.

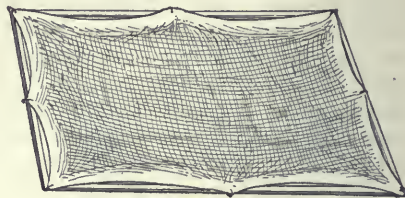


Fig. 124.—Strainer on frame.

to dispense; hence, in most pharmaceutic operations filtration is far preferable.

FILTRATION

Filtration is a nearly perfect variety of straining; in both processes the operation of clearing the liquid being purely mechanical. As an illustration bringing this forcibly to mind, let us imagine we have a ditch of dirty water in which are pieces of wood (large and small), waste paper, and suspended mud. The ditch narrows to an underground drain, and, in order that the drain does not become clogged up, a grating is placed at its entrance. This grating is perhaps only six or seven iron bars, but as the water passes by them, some of the wood and paper, not able to pass through the spaces, remains on the grating. Suppose we take that water which has passed through the grating into the drain-pipe and examine it. It will be found as dirty as before, save that the larger chips and the masses of paper are removed. It contains bits of straw, slivers of wood, and fragments of paper pulp that were small enough to pass through the grating. This unsightly mixture we pour on a strainer of cheese-cloth. The meshes in this are much smaller, of course, than the spaces in the grating, and the liquid emerges from the strainer much clearer, the strainer having kept back straw, wood-fiber, and paper pulp, along with considerable of the muddy sediment. However, it is still far from clear; the suspended mud in very finely divided form having passed through the meshes of the cheese-cloth, which were not quite small enough to retain the minute particles. So we take the cloudy liquid and pass it through filtering-paper, the meshes (the pores) of which are so small as hardly to be seen with the naked eye. This process usually completes the work of removing the undissolved particles, the liquid emerging from the filter perfectly clear.

Let it here be said that modern sanitary engineering has shown that filtration of water through long layers of sand removes not only visible dirt, but can, if properly carried out, remove bacteria of disease. Notable among such efforts are the water-works systems of Altona, Germany, and Lawrence, Massachusetts.

The substance which removes the impurities is called the filtering medium, and the clear liquid, the *filtrate*.

Apparatus used in filtration consist of the bottle or other container

into which the filtrate drops and the container holding the filtering medium. The latter in small operations consists usually of a funnel, while in large processes of filtration it consists of earthenware crocks, or tanks of iron, wood, or masonry. The latter being beyond the province of this work, reference will be had solely to *funnels*.



Fig. 125.—Plain funnel.

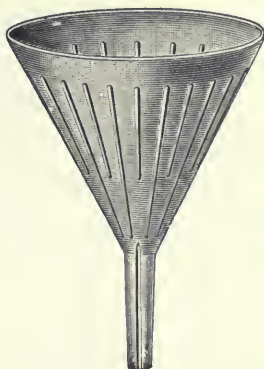


Fig. 126.—Ribbed funnel.

Funnels consist of conic vessels of glass, porcelain, or metal, although the latter should be carefully avoided in delicate pharmaceutical operations. To secure the best success from a funnel, it is necessary that the cone shall be at an equilateral triangle, as otherwise a folded filter-paper will not accurately fit same. Of the many varieties of funnels suggested for pharmacists' use, reference can be had here merely to a few types. It has been the writer's experience that just as good results are obtained by using a plain glass funnel (Fig. 125) as can be secured with more elaborate varieties, the main point in successful filtration being the correct folding of the filter-paper. A very popular form of funnel is the one with the ribbed interior, to permit the passing down of the filtered liquid (Fig. 126). This, however, can be accomplished just as well by the plaits of a carefully folded filter.

The same funnel is provided with a groove on the under side of the neck of the funnel to permit the exit of air from the bottle into which the liquid is filtered. It is better pharmacy, however, not to place the funnel directly in the neck of the receiving bottle, but to support it on a ring of the retort stand (Fig. 127).

If circumstances are such that the funnel must be placed in the neck of the bottle, some provision for access of air must be made, and this is done by interposing between the neck of the funnel and that of the receiving bottle a piece of cord; otherwise the air within the bottle becomes compressed as the liquid passes into the bottle until either the liquid ceases to filter through or the air, in forcing its way out, pushes

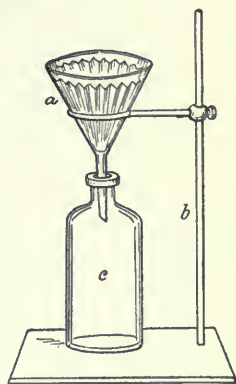


Fig. 127.—Correct filtration: a, Funnel; b, filtering stand; c, receiving bottle.

with it a certain quantity of the filtered liquid to the outside of the receiving flasks, producing an untidy operation.

This is particularly the case if the neck of the funnel is either too large or too short. It should enter completely the neck of the bottle and pass within the wider portion of same.

The **filtering medium** consists of paper, cotton, asbestos, sand, or stone, the first two being generally used in retail pharmacy.

Paper filters consist of sheets of unsized paper, which, when properly folded, form a conic bag on which the liquid to be filtered is poured. In commerce we find two varieties of such filter-paper, one being gray and the other white. The latter is more expensive than the gray, but should always be used, as the gray is made from common unsterilized rags, and is loaded with bacteria, whose least deleterious work is rendering the filtered liquid prone to fermentation or other decomposition.

The filter-paper can be folded in two ways (usually termed *plain* and *plaited filters*), and each has its peculiar use, plain filters being indicated when the precipitate is desired, and plaited filters when the filtrate is sought for.

In making a plain filter, the paper is folded but twice, in each case the sheet being divided exactly in half. The result is a perfect cone, with one thickness of paper on one side and three thicknesses on the other, which naturally produces unequal filtration. In order to equalize, we very frequently use two such plain filters, so alternating that both sides will be four layers of the filter-paper. At its best a plain filter does not remove the liquid with rapidity because the paper lies flat against the surface of the funnel, and the only place through which the filtrate can readily pass is at the point which lies exposed at the neck of the funnel. Therefore, for rapid filtration, a plaited filter is best.

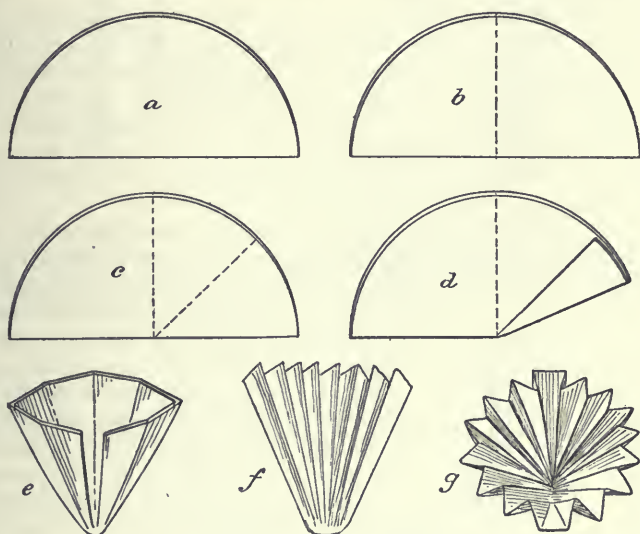
In the plaited filter the paper is so folded as to make a cone of equal thicknesses on all sides, and is also provided with canals through which the filtrate can run. The plaiting of a filter is a matter of considerable skill, and there is but one correct way to fold a successful filter. To thoroughly understand the method it is necessary to accompany the explanation by illustrations (Figs. 128 and 129). The circular piece is folded exactly in the center (*a*). This semicircle is again folded in its center (*b*). The semicircle is then opened and half of the semicircle is folded upon the central crease (*c*). This gives one-fourth of the circle divided into eighths, and these eighths are now folded into sixteenths (*d*), each fold being against the upper surface of the filter-paper.

Attention is then paid to the other fourth of the circle, it being folded exactly as was the first fourth, and as a result it being folded into sixteenths as shown in (*e*). These sixteenths are then divided into thirty-seconds by folding each back on itself, and when finished the unopened filter should present the appearance of (*f*), that is, a series of perfectly symmetric plaits, only two of which will rest against the edge of the funnel, and these can be removed by folding same into sixty-fourths, as (*g*). The observance of the following details will contribute to attaining the ideal in filtration:

First, the folds of the filter should not be permitted to extend to the point of the filter, but in plaiting the filter the crease should be made from the point toward the edge, a finger being kept on the point to prevent the creasing of same. The necessity of sparing the

point can be understood when it is appreciated that every crease in the piece of paper contributes to the weakening of same, and, therefore, the passing of sixteen creases across the central point forming the tip of the filter is apt to so weaken it that it will break as soon as the liquid will be poured upon it, the greatest pressure of the liquid being exerted on the filter tip.

Second, the filter should be moistened before filtering. This opens the pores of the filter, rendering easier the passage of the liquid through the filter. It is needless to say that the filter should be moistened with the liquid which is the solvent for the drug intended to be filtered. Thus, to moisten with water a filter through which it is intended to pass essence of ginger, would be doing much more harm than good. The moistening of such a filter should be done with alcohol.



Figs. 128 and 129.—Folding plaited filter (see text).

Third, when the filter is moistened, it is advisable to push the tip of the filter fairly well down into the neck of the funnel. This serves to strengthen the filter tip.

Fourth, in pouring the liquid on the filter, avoid throwing it directly on the tip, but gradually pour it upon one side of the filter, as otherwise the force of the fall is apt to rupture the paper.

Fifth, the paper should not project above the funnel. It is a human failing to fill the filter as completely as possible, and in case the paper projects over the side of the funnel, the actual level of the liquid may be above the top of the funnel, and a rupture of the paper brings disastrous results. The chief reason, however, for this rule is that the projecting paper affords considerable surface exposed to the air, thereby producing spontaneous evaporation, with accompanying loss of the liquid. This is particularly the case when the liquid to be filtered is volatile.

A convenient application of paper as a filtering medium is seen

in the Wetmore filter, which can be used either by pressure or syphoning.

Cotton Filter.—In many cases of simple filtration all that is necessary to remove the insoluble matter is the passing of the liquid through a pledget of cotton placed in the neck of the funnel. This method of filtration is one which should be practised more frequently behind the prescription counter than is usually the case. Every prescription calling for a solution should be dispensed absolutely clear, and to insure this the finished solution should be filtered through cotton before dispensing. If three or four funnels of various sizes be kept within reach and a package of absorbent cotton be kept in the drawer of a prescription case, such filtration is exceedingly simple, the custom of the writer being to filter directly into the bottle in which the prescription is dispensed.

Asbestos Filter.—Instead of a plug of cotton, a similar plug of purified woolly asbestos inserted in the neck of the funnel affords an admirable method for the filtering of strong acid or alkaline liquids, which would destroy filter-paper or cotton. Such asbestos wool can now be easily obtained at moderate cost, and should share a place in the drawer in which the absorbent cotton is kept. By means of the asbestos it is a very simple matter to filter a solution of soda or solution of potassa.

Sand- and stone-filters are used in large operations only, such as filtering drinking-water. The sand-filter usually consists of a vat in which are placed alternate layers of sand, charcoal, and gravel, usually to a depth of two or three feet. Upon this the water is thrown and percolates through to the bottom, and is drawn off by means of a faucet.

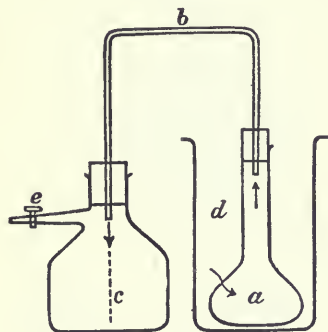


Fig. 130.—Porous flask filter

Many patent varieties of stone-filters are in the market, they consisting of porous stones cut into shapes suitable for the container, and through which the liquid passes. A very convenient and small modification of the stone-filter is the porous flask (Fig. 130), which is attached to a syphon and placed in a vessel containing the liquid desired to be filtered. The play of the syphon once established, the liquid passes from

the original container through the porous flask, and emerges from the syphon completely filtered.

Volatile Filtration.—In case the liquid to be filtered is volatile, precautions must be taken to prevent undue evaporation. This is usually accomplished by placing the filter-paper in a funnel considerably larger than itself, covering the funnel with a sheet of rubber or plate of glass, while the neck of the funnel fits into a cork stopping the neck of the receiving bottle. In order to permit exit of air from the receiving bottle and ingress of air into the top of the funnel, these two may be connected with a rubber or glass tube in the same way as explained under Volatile Percolation (p. 260).

Hot Filtration.—Some liquids can be filtered only when quite

hot, and with certain easily fusible solids the best method of purification is by filtering same in the molten state; such, for example, is the case with wax. In all such cases the funnel containing the filtering

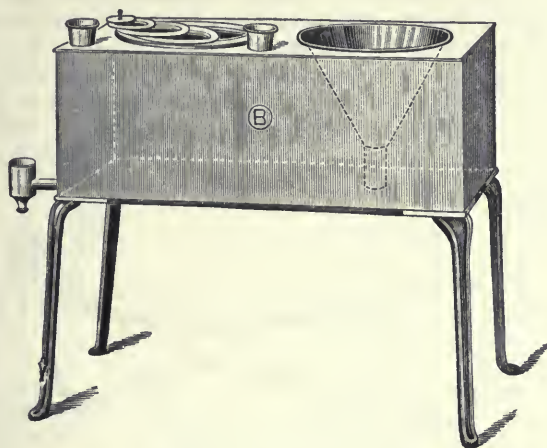


Fig. 131.—Hot filtration bath.

liquid must be kept warm, and for such purposes various forms of hot filtration apparatus have been devised (Fig. 131). These usually consist of water-baths with the conic holes in which the funnel will

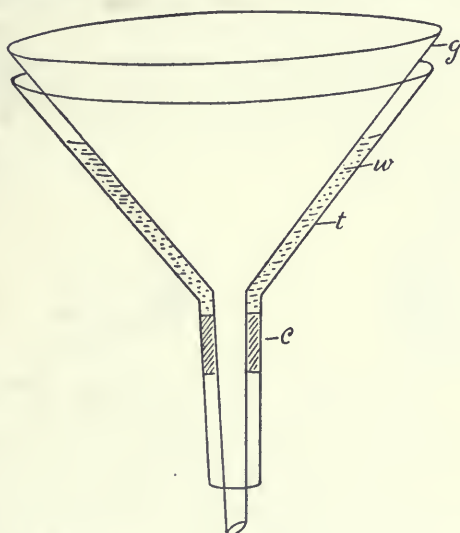


Fig. 132.—Funnel for hot filtration: *g*, Glass funnel; *t*, tin funnel; *w*, water; *c*, cork (Hatcher and Sollmann).

fit. A cheap and practical form of such hot filtration apparatus, devised by Dr. R. A. Hatcher, consists of a tinned iron funnel with its neck closed by a perforated cork, through which passes the neck

of a glass funnel. In the space between the inner wall of the metallic funnel and the outer wall of the glass funnel water is placed and kept warm by means of a Bunsen burner (Fig. 132).

Rapid Filtration.—In many cases of ordinary processes of filtration the liquid filters through so slowly that some means to increase the flow are necessary. This is usually accomplished by fitting the receiving bottle with a two-holed rubber cork, through one perforation of which the neck of the funnel passes, while the other is fitted with a bent glass tube connected with an apparatus which will rarefy the

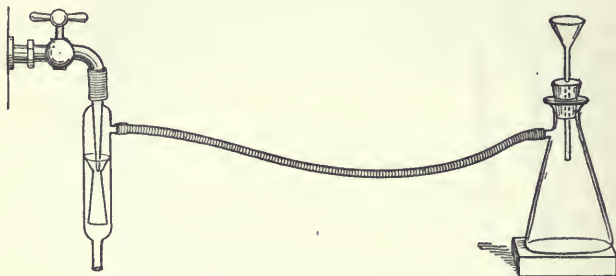


Fig. 133.—Rapid filtration.

air within the receiving bottle (Fig. 133). A special form of receiving bottle is obtainable in which the air is drawn off by a tube connected from the side of the bottle (Fig. 134). In such cases the pressure of the atmosphere acting on the surface of the liquid in the filter is greater than the pressure within the receiving bottle, and hence forces the liquid through the bowl of the filter into the receiving bottle, and that frequently with such great force as to rupture the filter-paper unless some means are taken for its protection. This is usually accomplished in filtering liquids in quantitative chemistry by fortifying the



Fig. 134.—Filtering flask.



Fig. 135.—Gooch crucibles.

tip of the filter-paper with a cone of perforated platinum. Such quantitative precipitates, however, can best be separated from the liquids in rapid filtration by use of the Gooch crucible filter (Fig. 135), which consists of a porcelain crucible provided with a perforated bottom in which is inserted a plug of asbestos, and this, by means of the large rubber tubing, fitted to the smaller funnel connected with filtering flask (Fig. 136).

The rarefaction of the air within the flask can be accomplished with the water-pump pictured on p. 92, or, in its absence, sufficient air can be removed by the simple expedient of syphon suction.

The apparatus consists of two one-gallon jugs, each fitted with two-holed corks. Through the orifices of each cork passes bent glass tubing, one extending to the bottom of the jug, the other only a few inches below the under side of the cork. In the operation one jug is filled with water, placed on a table, and connected with the other jug placed on the floor by rubber tubing attached to the long

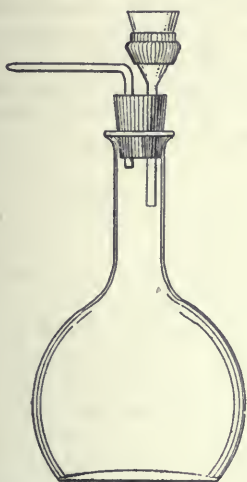


Fig. 136.—Gooch crucible in filtering flask.

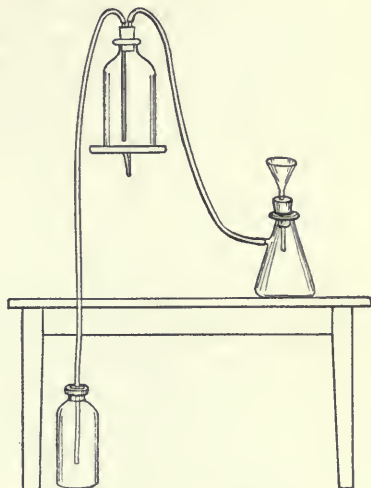


Fig. 137.—Syphon filtration.

glass tube of each jug. The small glass tube of the upper jug is fitted by thick rubber tubing to the end of the aspirator, and the water in the jug is made to syphon over into the lower jug, thus producing a suction of the upper jug and the filtering bottle. When all water has run into the lower jug, the position of vessels is reversed and the operation continued (Fig. 137).

CHAPTER VIII

CLARIFICATION, DECOLORIZATION, AND SEPARATION OF IMMISCIBLE LIQUIDS

CLARIFICATION

CLARIFICATION is the process of clearing a liquid without the aid of filtration. As filtration is a simple and thorough operation, clarification is substituted for it only in cases where filtration is difficult, if not impossible, as in the case of mucilaginous and other viscid liquids which will not pass through the pores of a filter. The clarification of liquids, whenever possible, is accomplished by means which are purely mechanical, clarification by means of chemicals not being

recommended because of danger of alteration of the liquid during the process. The apparatus for clarification is usually of the simplest character, consisting generally of a vat, kettle, or dish, and some form of strainer through which the liquids are passed to remove it from the solids. Clarification is accomplished in the following ways:

1. *Heating*.—In many cases the liquid to be clarified is turbid by reason of suspended or partially dissolved albumin or wax. In such cases heating usually accomplishes clarification by coagulating the albumin or by causing the waxy substance to liquefy and rise to the surface. After such cases of clarification the cool liquid is either strained, or the separated solids float on the surface (scum) and are removed by skimming. An illustration of this kind of clarification is the process of clarifying honey, as given in the pharmacopœia of 1880.

2. *Increasing Fluidity*.—In some cases suspended particles of dirt, grit, and straw make turbid a liquid which is so viscid that these particles can neither rise to the surface of the liquid if lighter than same, nor sink to the bottom if heavier. This can be remedied by diluting the liquid with water until sufficiently limpid to permit separation of the solid matter and then evaporating the excess of water.

3. *By Use of Albumin*.—The method given above is not always satisfactory, as frequently it is inconvenient to evaporate the excess of water, and, moreover, it is often the case that the particles do not separate of their own volition. The separation of these particles can be facilitated by thoroughly mixing white of egg with the liquid and then heating the mixture, when the albumin coagulates and rises to the surface of the liquid, carrying with it the mechanical impurities, which are skimmed off along with the solidified albumin. By this process boiled coffee is rendered clear.

4. *By Use of Milk*.—In the above operation milk is sometimes substituted for albumin, this acting the same way by reason of the coagulation of the casein it contains.

5. *Paper Pulp*.—A similar mechanical separation of the particles can be accomplished by tearing filter-paper into shreds, mixing it thoroughly with the liquid, and heating. When the paper rises to the surface it carries with it the suspended particles, which are removed by skimming. The official method of clarifying honey combines the use of paper pulp with increasing the fluidity of the liquid.

6. *By Sedimentation*.—If the particles rendering the liquid turbid are sufficiently heavy, they will settle to the bottom of the containing vessel, even though the liquid is quite viscid, if sufficient time is given to accomplish the separation. This slow method of separation is called *sedimentation*, and the insoluble matter finally settling at the bottom of the vessel is called the *sediment*. The term *precipitate*, frequently applied to the sediment, is incorrect, as by precipitate we mean insoluble matter which has been in solution, whereas the particles composing sediment have never been dissolved.

7. *By Use of Gelatin*.—Gelatin is only employed for clarifying liquids containing tannin, the action in this case being chemical, and not mechanical. Gelatin forms with tannin an insoluble combination, similar to leather.

8. *By Fermentation*.—This is a method of clarification, the appli-

cation of which is limited to the preparation of fruit-juices used at soda fountains. The preparation of these is a very simple matter. Fruit is crushed either by pounding in a tub or by means of a fruit-crusher or grater, and the pulp allowed to stand in a comparatively warm place for twenty-four hours. Partial fermentation is begun, and the slight change in the chemical character effects a separation of the clear juice from the pulp and albuminous matter, permitting the easy separation of the juice from the insoluble matter by straining, and the strained juice is then placed in bottles, corked, and sterilized by submitting to the action of boiling water for half an hour or more, or, if preferred, the sterilization can be omitted and the fruit preserved by the addition of alcohol. Some of the commercial juices are preserved by the addition of salicylic and other preserving agents, but in most States the addition of these preserving agents is prohibited by legislative action.

DECOLORIZATION

Decolorization is the process of removing color from a substance, and is usually accomplished by macerating the solution with animal charcoal for several days and then filtering. Animal charcoal is the only convenient substance possessing this decolorizing property, due to the powers of absorption it possesses. This absorbing ability is not limited to coloring-matter, but also to glucosides, alkaloids, and bitter principles, hence in decolorizing decoctions of the drugs containing the latter substances by animal charcoal they suffer some loss in active principles.

SEPARATION OF IMMISCIBLE LIQUIDS

By immiscible liquids we mean two liquids which do not dissolve in each other, for example, water and oil, and in cases where these two are brought together, it is very frequently useful to have a means of separating them readily. This separation is accomplished in several ways. In case the amount of one of the liquids is very small, it can be separated from the other by means of a *pipet*, one end being placed in the liquid, which is drawn into the pipet by means of suction (Fig. 138).

A similar form of crude separation can be accomplished more thoroughly by a cheap glass *syringe*, the suction in this case being accomplished by moving the piston. Both these means of separation, however, are very primitive, and should be used only in emergencies. Absolutely thorough separation can be accomplished by the inexpensive *separatory funnel* (Figs. 139 and 140), which consists of a glass tube provided with a glass stopper and terminating in a tapering tube to which is fitted a glass stop-cock. For those economically inclined, an ordinary glass funnel can be used as a separatory funnel by fitting in the neck of same a velvet cork, fitting the glass rod, and allowing the liquid to run out from the funnel by raising the cork and pressing it down until the last portion of the heavy liquid has been separated. Considering, however, the low price of a separatory funnel, the use of this crude apparatus is hardly justified.

One immiscible liquid can be crudely separated from another by

means of a *syphon*, but to this method the same comments can be applied as to separating with a syringe.

By Florentine Receiver.—For the separation of a quantity of immiscible liquids too large for the separatory funnel, recourse is had to the Florentine receiver (Figs. 141, 142, and 143), these being used in the steam distillation of substances containing volatile oils to separate the volatile oil from the water which accompanies it in the

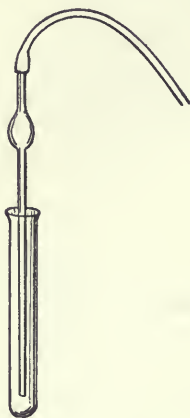


Fig. 138.—Separation of immiscible liquids with pipet.



Fig. 139.—Separatory funnel.



Fig. 140.—Squibb's separatory funnel.

distillation. As shown in cuts, two forms of such receiver are used, accordingly as the oil is heavier or lighter than water, each apparatus being so arranged as to cause the flowing out of the water and retention of the oil in the receiver, while the third cut shows a receiver adapted to either light or heavy oils.

A crude method of separating chloroform from large quantities of water, such as is sometimes required in the extraction of alkaloids

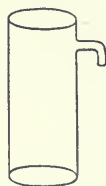


Fig. 141.—Florentine receiver for heavy liquids.



Fig. 142.—Florentine receiver for light liquids.

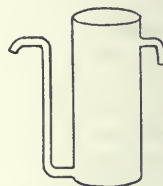


Fig. 143.—Florentine receiver combined.

from drug decoctions by shaking with chloroform, is by agitating the mixture in a five-gallon demijohn or can, and suddenly inverting same into a quart or half-gallon wide-mouthed bottle, and when the chloroform has completely separated into the jar, the demijohn is removed and the chloroform separated from last small quantities of water by use of an ordinary separatory funnel.

CHAPTER IX

PRECIPITATION, CRYSTALLIZATION, GRANULATION,
EXSICCATION, AND DIALYSIS

PRECIPITATION

PRECIPITATION is the process of separating a solid from its solution by the aid of physical or chemical action. The substance so separated is called a *precipitate*; the chemical producing this precipitate is called the *precipitant*, while the liquid remaining above the precipitated substance is termed the *supernatant liquid*.

As mentioned in the last chapter, a precipitate differs from a sediment in that a precipitate is separated out from its former state of solution, while a sediment has never been dissolved. In order to precipitate a substance it is necessary to first get it into solution. On the other hand, the precipitated substance is, in the nature of things, a substance insoluble in water, and must, therefore, be dissolved by special agents, the act of solution frequently bringing about a chemical change.

Precipitation may be due to chemical or physical causes. The most important phase of precipitation is that involving chemical action, wherein the two reacting substances are dissolved in separate portions of water, and on mixing these solutions an insoluble body will be formed and precipitated.

But we have precipitations that are due to purely physical causes. Thus from a hot supersaturated solution on cooling will be precipitated the excess of dissolved substance. Likewise, solution of such substances which are more soluble in cold water than in hot (like lime) will be precipitated when they are heated, such cases being examples of precipitation through change in temperature. We have learned that substances may be soluble in one liquid and insoluble in another; thus, gum arabic dissolves in water, but is insoluble in alcohol. If alcohol is added to a solution of acacia, it diffuses through the water and the acacia is precipitated, an example of precipitation through change in menstruum.

Objects of Precipitation.—We precipitate substances chiefly to *purify same*. As will be noted later, the purification of chemicals which are soluble is easily effected by crystallization, and, likewise, the purification of insoluble chemicals is generally accomplished by precipitation, and in both cases the act of purification is accomplished by bringing the substance into solution. Therefore, in precipitation we dissolve a substance in some way usually by changing the identity of the chemical. The solution thus obtained is rendered free from insoluble extraneous matter by filtration, and to the filtered liquid is added the appropriate precipitant, which converts the chemical into the insoluble form desired, leaving the soluble impurities in the supernatant liquid.

Another object of precipitation is to *obtain the substance in fine powder*, the insoluble matter usually separating out in almost molecular subdivision.

A third object of precipitation is for *its value in chemical testing*.

Practically the entire scheme of analytic chemistry (Chapter LII.) is based on the separation of the various elements from solution by the addition of appropriate precipitant.

Operation.—As mentioned above, precipitation can be accomplished by either physical or chemical means, although by far the greater number of precipitates used in pharmacy and chemistry are produced by chemical means. Among the physical means of precipitation are by use of *heat*, as shown by the precipitation of the egg-albumen from the mixture by heating when the albumen coagulates, as explained in the preceding chapter.

Another physical form of precipitation is *by changing the nature of the solvent*. As mentioned in Chapter VII., a substance may freely dissolve in one solvent, but be insoluble in another, and in such cases the addition to the solution of the substance of the liquid in which it is insoluble usually effects the precipitation of the solid, provided always that the added liquid is freely miscible with the liquid which holds the salt in solution. A case of this kind is that of acacia, which will be precipitated from its aqueous solution on the addition of alcohol. This separation, it will be noted, is purely physical, and it differs from the precipitation of acacia from its aqueous solution by the addition of lead subacetate, in which case chemical change is effected.

Among the chemical means of precipitation it is possible to include the action of *light*. Even that precipitation is frequently considered as a physical means, but it is true that precipitation effected by light is brought about by the light changing the chemical character of the precipitated substance. One case where light produces precipitation is with the salts of silver. Almost every pharmacist has noted the precipitation occurring in solution of silver nitrate exposed to the light when the sides of the vessel become coated with a black substance, which is metallic silver, the light acting on silver nitrate, reducing it to the metallic form. The usual chemical method of precipitation, however, is *by adding a precipitant*, as mentioned above, and as illustrated in the manufacture of precipitated calcium carbonate from prepared chalk.

These two substances are chemically identical, the only difference being that prepared chalk—a natural product—is only slightly purified by the process of elutriation and trochiscation; hence is apt to contain more or less impurities, and it is freed from these impurities by the process of precipitation.

In converting the prepared chalk into calcium carbonate, five stages in the operation may be noted. The first is to get prepared chalk or the calcium part of it into solution, for it is through solution that a substance can be readily freed from impurities.

To effect this solution a chemical change is necessary; in other words, the insoluble calcium carbonate must be converted into a soluble salt of calcium, and the salt selected is calcium chloride. This calcium chloride is produced from the prepared chalk by the addition of hydrochloric acid, the reaction being:



. The calcium chloride solution so obtained is filtered to effect a

separation of insoluble particles, and this filtration might be considered the second stage of the process. The third stage of the process is the real precipitation, which is accomplished by adding to the solution of calcium chloride the precipitant, which is a solution of any soluble carbonate, and for practical purposes sodium carbonate is always used, the reaction at this stage being:



It will be seen that, besides calcium carbonate (precipitated chalk), sodium chloride is formed, and the moist precipitated carbonate is wet—not with pure water, but with a solution of salt. If we dry the precipitate just as it separates from the liquid of which it was once a part, we get a product contaminated with salt, but if we wash it, the sodium chloride dissolves, leaving behind pure calcium carbonate moistened only with water.

Therefore, we wash the moist precipitate, this constituting the fourth stage in the process of precipitation. Lastly, the moist precipitate must be dried, which constitutes the fifth and last stage of the process.

Apparatus used in precipitation by chemical means consist of a chemical flask or evaporating dish in which the insoluble substance is made into the soluble form, the funnel and filter by means of which the solution is rendered clear, the jar or dish in which the filtered solution is mixed with the precipitant, and where the precipitate is allowed to collect, a strainer or a plain filter in which the precipitate is collected and washed, and, lastly, a drying closet or other means of desiccation by which the moist precipitate is dried. All these pieces of apparatus have been considered except the precipitation jars; these consist either of tall cylinders or of beakers, both of which were illustrated under Decantation (Figs. 119, 120). The precipitating jar is of glass, made tall in order to effect the complete separation of the precipitate, thus facilitating decantation; the beaker is a vessel of thin glass so annealed as to stand heating.

For precipitation in chemical testing, test-tubes are usually employed. These consist of narrow cylinders of glass, five or six inches long, with round bottoms

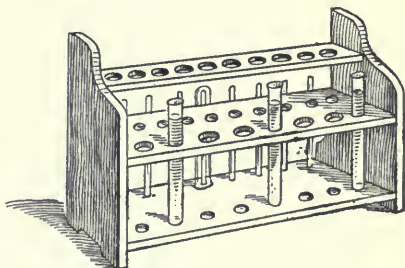


Fig. 144.—Test-tubes in rack.

(Fig. 144), while in quantitative analysis the precipitates are carefully collected in a beaker, as explained under Decantation.

The appearance of the freshly separated precipitate is described by appropriate adjectives, most of which are self-explanatory. Thus, we say that silver chloride is *curdy*; ammoniomagnesium phosphate is *crystalline*; aluminum hydroxide is *gelatinous*; coagulated albumin is *flocculent*, and magnesium phosphate is *granular*.

Sometimes, when a precipitate is light and occupies considerable space, it is said to be *bulky*. A *magma* is a form of thick, tenacious precipitate, such as is formed by the official ferric hydroxide.

In explaining a test, mere mention of the precipitant is not suffi-

cient, but the color and character of the precipitate formed should be included in the answer. Thus, if asked the test for sulphuric acid or a sulphate, it is scarcely complete to answer "barium chloride," but such a question should be answered: "Barium chloride gives a white precipitate, insoluble in nitric and hydrochloric acid." The last part of this answer, while seemingly superfluous at first glance, is necessary, inasmuch as barium chloride forms white precipitates with several other acids, but it is only in the case of sulphuric acids and sulphates that the precipitate is insoluble, as mentioned.

In some cases the same substance can be precipitated in a light, flocculent form or in a heavy, granular form, giving rise to the distinction between *light* and *heavy precipitates*. These result entirely from the method of manipulation. If, in precipitating the substance, the solutions are dilute and cold, a light precipitate is usually formed, whereas if the two substances are mixed in hot concentrated solution, a heavy precipitate is formed. An official illustration of these two forms of the same substance is shown in magnesium oxide, U. S. P., and heavy magnesium oxide, these being produced by the calcination of light magnesium carbonate and of heavy magnesium carbonate respectively, the light being precipitated from dilute solutions and the heavy from concentrated solutions.

CRYSTALLIZATION

Crystallization is the process of separating substances in forms possessing definite geometric angles. Such substances are called "*crystalline*," while substances which cannot be made to form definite geometric angles are said to be "*amorphous*." Among the latter are some substances which are sometimes erroneously considered crystalline, as, for example, the scale salts of iron.

In considering crystallization the first aim is to get an idea of what a crystal is. A crystal is a solid whose faces form definite angles, a body which can be measured according to the rules of geometry. Geometry is that portion of mathematics which deals with the measurement of surfaces and angles. Angles are formed where surfaces meet and vary in size, being measured by their relation to the circle. Thus, were a right angle cut from a circular disk from center to edge, it would remove one-quarter of the circle. A circle in geometry is divided into 360 parts, called degrees, and since the right angle is one-quarter the circle, its measurement is said to be 90 degrees. An angle of 60 degrees is but one-sixth the circle, hence is sharper than a right angle.

An angle means what we usually call a corner—a space inclosed by the intersection of two straight lines. We have instruments whereby these angles may be measured, and the measurement of the various angles forms a part of the sciences of geometry and trigonometry. Among the practical applications of these sciences may be mentioned surveying and measurement of crystals. In physics and astronomy a knowledge of these sciences is essential.

Crystals are specially interesting as definite geometric forms assumed of themselves without outside aid. It is an easy matter for a skilled sculptor to hew a definite geometric form, but remember that a crystal form depends on no human aid. The substance com-

posing the crystal groups its particles into the shape ordained for it from the beginning, and every perfect crystal of the same substance, be it large or small, has exactly the same form. Then, too, all substances do not crystallize with the same angles. One for ages has been crystallizing in a certain form, while one of its neighbors during the same period has produced numberless crystals all alike, but each of shape different from the substance first spoken of. In this way we have in the study of crystals an excellent method of distinguishing different chemical substances, and so large is the scope of the work that crystallization is now classed as an independent science.

In studying the crystal the crystallographer distinguishes four different parts to a crystal, the most important of which are the *angles* described above, an angle being the measurement of the conjunction of two *faces*, such conjunction being called the *edge*.

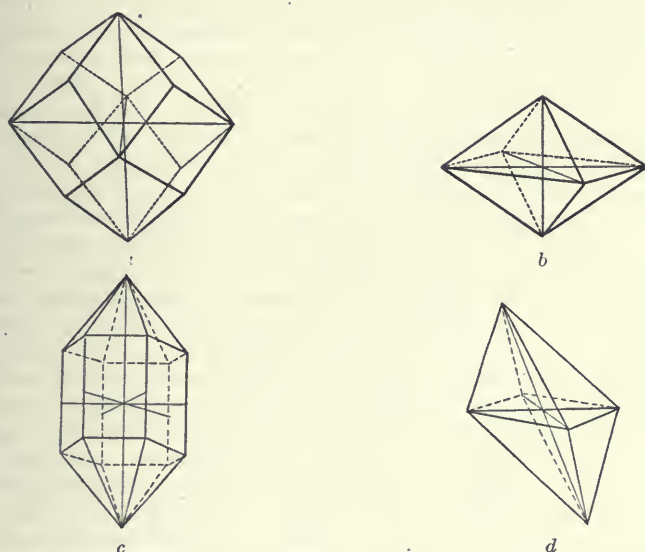


Fig. 145.—Crystal forms: *a*, Dodecahedron; *b*, quadratic octahedron; *c*, hexagonal prism; *d*, double oblique octahedron (Holland).

These terms will best be understood by a study of Fig. 145, which also shows the fourth characteristic of the crystal, the *axes*, these being imaginary lines so drawn through the crystal that they intersect at its center. Each crystal has its peculiarities of faces, axes, and angles, and, therefore, all substances in crystalline form are grouped by crystallographers into various crystal systems, the main division being as follows:

NUMBER OF AXES.	LENGTH OF AXES.	ANGLE OF INTERSECTION.
Regular.....3	All equal.	All right angles.
Tetragonal.....3	Two equal, 1 different.	All right angles.
Rhombic.....3	All unequal.	All right angles.
Hexagonal.....4	Three equal, 1 different.	Three at angle of 60 degrees, the fourth at right angle.
Monoclinic.....3	All unequal.	Two oblique, the third at right angle.
Triclinic.....3	All unequal.	All oblique.

The subject of crystallography is beyond the scope of the average pharmacist, its chief value being as an adjunct to the science of mineralogy, therefore, the rest of this chapter will be devoted to the pharmacæutic consideration of crystallization, and those desiring further information on the science of crystallography are referred to standard books on the subject.

This is following the lead of the pharmacopœia, which rarely mentions the scientific classification of crystals, contenting itself with the rather superficial general terms applied to crystals, such as saying that the crystal is "prismatic" (in shape of a prism), "tubular" (somewhat round cylinder), "acicular" (needle-shaped), etc.

In some cases mineralogists find half-crystals; in other words, the ideal crystal is split down its length. Such crystal splitting is called *cleavage*.

Isomorphism.—Substances which crystallize into the same form are said to be *isomorphous*; and, through the investigation of Mitscherlich, has been evolved the interesting and valuable theory that isomorphous bodies usually possess closely allied chemical composition. Thus, sodium phosphate, $\text{Na}_2\text{HPO}_4 + 12\text{H}_2\text{O}$, and sodium arsenate, $\text{Na}_2\text{HAsO}_4 + 7\text{H}_2\text{O}$, are found to be isomorphous. In the same way the alums all crystallize in the same form.

Measurement of Crystals.—As mentioned above, crystallography is the study and classification of crystals chiefly according to the angle intersection, the measurement being made by the instrument called *goniometer*. A full description of this instrument would be too technical for the limits of this work, but its principle of action can be explained by saying that in this instrument the crystal, the angles of which are desired to be measured, is placed in an appropriate holder, operated on a universal joint, capable of rotating in every direction, and the angle of rotation can be carefully measured on an appropriate scale. A telescope is part of the instrument, and through this we gaze on an image reflected from one side of the crystal, and we then gently rotate the crystal, when the image becomes lost, until the rotation has brought the adjoining face of the crystal into exactly the same position formerly occupied by the other face, when the image is forthwith restored. At this point a reading is made of the rotation of the crystal, and from this datum the angle can be determined.

Objects of Crystallization.—The pharmacist's consideration of crystallization is limited almost entirely to the production of crystals, and, therefore, the view of the pharmacist as to crystals is quite different from that of the crystallographer, the latter busying himself with the examination of the finished and usually native crystal, while the aim of the druggist is the production of a handsome crystal without special reference to the exactness of the angles. He produces a substance in crystalline form, but merely to obtain them in a higher degree of purity, and to this he adds the esthetic consideration that a perfect crystal is the most beautiful form in which a chemical can be produced.

For practical reasons, however, the pharmacist usually prefers his chemicals in the form of granular crystals, rather than a large and perfect sample, the former being readily more soluble.

Production of Crystals.—The several ways in which crystals are produced are given place in order of their relative importance:

First, by cooling a hot saturated solution. This is the usual method of producing crystals in chemical manufacture, and the process can be readily understood by one who has studied the preceding chapter on Solution. Therein it was noted that a substance is usually more soluble in hot water than in cold; furthermore, that from this hot solution, on standing, will be deposited the excess of the substance.

Thus, as cited on p. 120, 100 Cc. of boiling water will dissolve ten times as much potassium chlorate as will 100 Cc. of water at 15° C., and when the temperature of this heated solution is reduced to 15° C., the 90 per cent. excess will separate out, and, indeed, in the form of crystals, since the body is crystallizable. Such is the usual method of making crystals, the substance being dissolved in hot water, the hot saturated solution filtered through paper, the filtrate allowed to stand in an appropriate dish, usually for twenty-four hours, when the liquid is separated from the crystals, which are then removed from the dish and dried.

Two technical terms are used in connection with this process. The liquid above crystals in which the substance has formerly been dissolved is called the *mother liquor*, and, again, in some cases of crystallizing this way a dilute solution is concentrated by evaporating until a thin scum of crystals forms across the surface of the liquid, and this layer is called the *pellicle*.

Apparatus.—The apparatus used in this operation are the indispensable funnel and filter and the dish in which the crystals are collected. For small operations no better dish can be found than the ordinary pint evaporating dish. Glass beakers are sometimes used, but should be employed only for the collection of minute quantities, and when the crystals are not particularly large and strong, as in the latter case the removal of the crystals from the sides of the vessel is liable to result in a fracture of the glass. In all operations of crystallization crystals will separate out more readily if the interior of the dish is rough, although this detail is valued chiefly in crystallizing large quantities, for which purpose nothing is better than the ordinary earthenware crocks. The roughness of the vessel seems to attract the formation of crystals, the same way as does the introduction into the liquid of bits of glass or tightly stretched twine.

These extraneous aids to crystallization are called *nuclei*, and a familiar illustration of nuclei is the use of string on which rock-candy crystallizes. The finished crystal must be drained from its mother liquor, and this is usually accomplished by throwing the wet crystal mass on a strainer, or, in the case of smaller quantities, on a filter, and allowing it to drain. The excess of the liquid is then removed by placing the crystals between sheets of filter-paper, or, in the event of the substance being of a caustic nature, such as chromic acid, porous plates of unglazed earthenware are used.

Second, by the gradual evaporation of a solution. This consists of a modification of the foregoing, the crystallizing solvent being in this case alcohol, ether, chloroform, or some other volatile liquid. In such cases the chemical (usually of plant origin) is dissolved in the smallest possible quantity of the liquid, the solution filtered into

an evaporating dish and allowed to stand, when, due to the gradual vaporization of the solvent, the crystals slowly form in the bottom of the dish. In such case the crystals should be removed before the solvent has completely dissipated, in order that the impurities may be removed in the solvent.

Third, Sublimation. Some chemicals, on subliming, solidify in the form of crystals. Such is particularly the case with benzoic acid, and this gives the method employed in the manufacture of this chemical from its natural source, the official benzoin. The drug is coarsely powdered, mixed with sand, and placed in a dish which is covered with a piece of cheese-cloth. Over the dish is placed a cone of paper, and the entire apparatus is placed on a sand-bath and heated at a temperature not exceeding 110°C . From the benzoin the benzoic acid is liberated in vapor form and rises within the cone, and is condensed by the lower temperature of the surrounding air in the form of beautiful crystals (see page 107).

Fourth, By fusion and partial cooling. This method of crystallizing is of little value to pharmacists, its only application being in the preparation of one form of sulphur crystals. In this operation a considerable quantity of the substance, say 200 grammes, is placed in a Hessian crucible and brought to a state of fusion. As the pellicle forms on the surface of the cooling mass a hole is punched through, and the remaining liquid carefully poured out. On completely removing the pellicle it will be found that the sulphur has deposited in the form of monoclinic crystals. In a similar manner Moissan prepared diamonds by melting iron and dissolving graphite therein, and allowing the mass to solidify, under great pressure, by plunging the white-hot mass into cold water. The solid mass is then treated with hydrochloric acid, which dissolves out the iron, when it is found that part of the graphite is converted into the crystalline form, diamonds; although the crystals are too small to possess commercial value. Perhaps Moissan's method is more properly considered as a case of deposition from saturated solution, as in truth the graphite is dissolved in the molten iron and is caused to separate out in crystalline form before the solvent has solidified.

Fifth, By effecting change in the character of the solvent. As mentioned under Solution, some substances are soluble in one solvent and insoluble in another. For example, sugar will dissolve freely in water and sparingly in alcohol. In this case, if to the concentrated solution of sugar is added alcohol in sufficient quantity, the sugar will separate out and, indeed, in the form of crystals. This method is used in effecting crystallization in many chemical operations, although its use in pharmacy is comparatively limited.

Sixth, By the electric current. This method is totally foreign to crystallization in pharmacy, and can be dismissed with the statement that handsome crystals of gold metal have been obtained by passing low currents of electricity through solutions of metal for long periods of time.

Water of Crystallization.—In the case of crystallization produced by a separation from a solution, the solvent very frequently plays a rôle in the composition of the deposited crystal, although in some cases the crystal separates from the solution without combining

with any of the solvent. The latter is usually the case when some solvent other than water is employed, but sometimes, in separating from aqueous solution, some chemicals form crystals that are water-free. If water combines with the separating crystal, as is usually the case in aqueous crystallization, it is designated as "*water of crystallization*," and if alcohol combines with the chemical in the same way, it is called "*alcohol of crystallization*," and in similar manner we find some crystals contain "ether" or "chloroform" or "acetic ether of crystallization," if these solvents have been employed in the process of making crystals. The last four cases are not usual, however, so our attention need be devoted to the water of crystallization.

By "water of crystallization" is meant water that is combined in some loose chemical way with the chemical of which the crystal is composed, and it does not mean the water adhering to a moist crystal. Thus, 47 per cent. of a perfectly dry crystal of alum consists of this water of crystallization.

The estimation of this water of crystallization is a very simple proceeding, all that is necessary being to heat a weighed quantity of crystals, first, on a water-bath, when it will be noticed the crystals liquefy, and then on a sand-bath, when the steam arising from the liquefied mass proves the presence of water. The heat being continued for a sufficient length of time, all the water will be removed, and a dried mass, known as the "exsiccated" salt, remains.

In the case of alum, 100 Gm. of the crystals dried with proper heat will leave but 53 Gm. of the exsiccated alum, showing that 47 per cent. of same is water. The amount of water of crystallization found in a given chemical is exactly the same for all perfect samples, and a study of the question has shown that the quantity of water in a crystal is in molecular proportion to the quantity of the substance, thus permitting the numeric expression of the amount of water in the chemical formula of the compound (see p. 378).

In this case, when we give the formula of sodium sulphate, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, we can deduce therefrom the molecular weight, and are brought to the conclusion that in every 278 Gm. of crystalline sodium sulphate there are 180 Gm. of water.

We can deduce in the same way from the chemical formula of potassa alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, that this contains 47 per cent. of water of crystallization, as explained on p. 535.

The term "water of crystallization" is gradually becoming obsolete, and instead we speak of the body containing such water as "hydrated." Thus, $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, is now called decahydrated sodium carbonate, while the official form, $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$, is called monohydrated sodium carbonate, and this term constitutes the pharmacopœial name of this salt. Recent research shows the difference between the decahydrated and the monohydrated forms of the same substance can be easily explained by the rules of physical chemistry; hence, the term "water of constitution," formerly used to designate the last remaining water, is being dropped.

Interstitial Water.—Among the chemicals which separate in crystal form from aqueous solution, without the absorption of water of crystallization, the best known example is common salt. This separates in handsome masses of cubical crystals, and, in separating, it very frequently mechanically closes certain quantities of water

which must be clearly differentiated from water of crystallization, because retained in a purely mechanical manner. Such water, which gathers in the interstices found in a mass of crystals, is called "*interstitial water*." On heating a substance containing interstitial water the latter is converted into a steam which, on expanding, tears the crystal mass asunder with a crackling sound. This phenomenon, called *decrepitation*, is different from the deflagration of potassium nitrate, which is due to the liberation of oxygen.

Efflorescence and Deliquescence.—The crystals of some substances, sodium phosphate, for example, part so readily with water of crystallization that even at ordinary temperatures the latter dissipates, causing the crystal to fall into a white powder. This loss of water of crystallization at ordinary temperatures is the phenomenon termed *efflorescence*. Special attention should be paid to the fact that an effloresced body means one which has lost its water of crystallization, and hence is stronger than the same body in the form of clear pure crystals. For this reason, in making a pharmaceutical preparation, care must be observed that, unless otherwise specified, the substance should be perfectly crystalline, and, if the effloresced salt is used in the same quantity as is directed for the crystalline, the finished preparation will be stronger than was intended.

Efflorescence, however, is not always loss of water of crystallization; thus when ammonium carbonate effloresces to ammonium bicarbonate, water of crystallization does not enter into the proposition (p. 490).

On the other hand, some crystalline substances have such affinity for water as to absorb same from the moist atmosphere, ultimately liquefying. Such substances are said to be "*deliquescent*," and a notable example of such deliquescence is the case of potassium acetate, which should, therefore, never be dispensed in papers.

Amorphous bodies and liquids which absorb moisture from the atmosphere in the same way are said to be *hygroscopic*. Thus, glycerin is a good illustration of a hygroscopic substance.

GRANULATION

Granulation is best defined as interrupted crystallization, while the operation of granulation is the process of heating a chemical substance with constant stirring until moisture is evaporated and a sabulous (coarse-grained) powder is produced. This definition, however, does not describe the manufacture of the so-called granular effervescent salts.

In granulation a substance is prepared as in crystallization, by separation from a supersaturated solution. As mentioned under Crystallization, to secure large and handsome crystals by this method it is usual to leave the hot filtered liquid perfectly quiet, a stirring of the same producing small, ill-defined crystals. As such is exactly the object of granulation, in that operation the hot filtered solution is stirred and the separation of the crystals facilitated by evaporation of the substance from the liquid to a point where a very small quantity of solvent remains.

In granulating a substance, however, the evaporation should not be continued until all the liquid has disappeared, as a certain quantity

of mother liquor is essential to remove soluble impurities by solution in the mother liquor.

Chemicals in granular form have of late years become quite popular, due to the more rapid solubility of same than the larger crystals, following the statement made under Solution that the latter is facilitated by comminution of the dissolving substance.

Apparatus.—In granulation the only apparatus needed are those required for crystallization from saturated solution, viz., flask in which the solution is performed, apparatus for the filtration, evaporating dish, and glass stirring rod. The operation itself will best be understood by preparing 20 Gm. of Granulated Ferrous Sulphate, U. S. P. (see Part VII).

Granular Effervescent Salts.—This class of preparations are totally different from the true granular salts, such as granulated ferrous sulphate, inasmuch as they are totally devoid of crystalline property, and are called “granulated,” because consisting of the chemical in uniform and small particles.

All these preparations are so made as to contain tartaric acid and some carbonate, so that when mixed with water copious effervescence occurs (p. 444). Here need it only be said that the preparations are made by mixing the active chemical with the acid and the carbonate, moistening with alcohol, and running the moist mass through a coarse sieve, whereby it separates into small and regular particles. The reason the mass is moistened with alcohol is that alcohol renders same sticky without producing the chemical action between the acid and the carbonate which would result were they moistened with water. For the same reason these preparations must be kept in tightly stoppered bottles to prevent contact with moist air, which would soon render them no longer effervescent. These salts can also be granulated by softening the mass by heat of a water-bath and passing the soft mass through a sieve.

EXSICCATION

Exsiccation is the process of removing water of crystallization from a chemical by the use of strong heat. This process was foreshadowed on a preceding page (p. 147), so here it need only be added that the chief object of exsiccating a substance is to reduce its bulk. Thus, a crystal of alum weighing 10 Gm. contains but little less than 5 Gm. of water, and but little over 5 Gm. of the chemical itself, hence 5 Gm. of exsiccated alum represents the same strength as about 10 Gm. of the crystal. In most operations the exsiccated salt has but little value over the crystalline, and perhaps is even less useful, as we usually employ chemicals in solution, and an exsiccated substance is more difficultly soluble than is the same substance in crystalline form. Therefore the chief use of exsiccated substances is for incorporation in pills, the size of which should be limited to 5 grains, and by use of 5 grains of the substance, represent about 10 grains of the same body in crystalline form. This explains the employment of dried ferrous sulphate instead of the crystalline, in making pills of aloes and iron. The chemical, dried alum, has, however, therapeutic value not possessed by the crystals.

Apparatus.—The apparatus used in exsiccation consists of an evaporating dish, which should be tared, while the source of heat is usually a sand-bath. (For details of operation, see Exsiccated Alum, Part VII.)

DIALYSIS

Dialysis or *diffusion* is a process whereby the crystalline portion of a mixture is separated from the non-crystalline by the passage of the former through a permeable membrane. The crystalline substance which passes through the membrane is called *crystalloid*, while the non-crystalline substances, usually of mucilaginous character, are called *colloids*, from the Greek "*kolla*," meaning glue.

The process of dialysis was discovered by Graham in 1861, and at the time of discovery it was thought it would be of unlimited use in pharmacy and other arts. Experience, however, has shown that its value is largely theoretic study of plant and animal physiology, proving that a large portion of the so-called vital processes going on in the living organism operates upon the principle of dialysis.

The apparatus used in dialysis consists primarily of any porous membrane. For this purpose a bladder is usually employed, although parchment paper or unglazed earthenware is employed for the purpose.

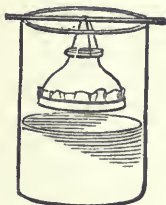


Fig. 146.—Dialyzer.

The rest of the apparatus consists of two containers of glass. The membrane is attached to a bottomless cylinder of glass or other suitable material, in such way as to serve as the bottom, and is made to rest upon the surface of water held in the second container, and upon the membrane is poured the liquid which is desired to be dialyzed (Fig. 146). This done, the crystalloids contained in the liquid on the dialyzer begin to pass through into the liquid below, whereas the non-crystallizable substance remains behind, thus

affording a very simple method of separating the mixture of, say, sugar and starch, the sugar being crystalline, passing through the membrane, and the starch remaining behind.

Objects.—As mentioned above, the most important object of dialysis is in the natural processes of animal and vegetable economy. Some soluble solids pass from one section to another by the process of dialysis. In the pharmaceutical application of dialysis a class of preparations, called "*dialysates*," were put upon the market. These are usually made by placing a decoction of plants on a dialyzer and evaporating the crystalloid liquid. The latter contained the crystalline alkaloid, whereas the residue left on the dialyzer consisted of the usually inert colloidal substance. As examples of such preparations occasionally used may be mentioned dialyzed opium and dialyzed cinchona, but these preparations did not come up to first anticipations, and at the present time practically the only dialyzed preparation used to any extent in pharmacy is dialyzed iron, which is a colloidal preparation. (See p. 564.)

In chemistry dialysis is of some value in the separation of crystalloidal substances from obnoxious colloidal matter. This is particularly the case in toxicologic examinations, when searching for

alkaloidal poisons; the latter can be separated from the stomach-contents by throwing the matter on a dialyzer and evaporating down the crystalloidal liquid. Even in this case, however, the application is somewhat limited because too slow, and other methods of separation are usually employed.

CHAPTER X

EXTRACTION

UNDER the head of Extraction are grouped the processes in which the active and soluble constituents of a drug are separated from the inert insoluble portion by the use of an appropriate solvent. These processes comprise maceration and expression, percolation, digestion, infusion, decoction.

In order to understand the principle underlying the process of extraction it is necessary to appreciate the structure of the ordinary vegetable drug. Any drug representing a plant part is comprised of a collection of cells, lately living or long dead, each one of which possesses a wall of more or less thickness, this wall consisting of some variety of cellulose, possibly lignin or suberous, corky tissue. This cell-wall is an insoluble thick envelop, scarcely permeable to liquid, while the active principles which we seek are generally found in the orifice encircled by the wall. To get the solvent in direct contact with the soluble constituents within the cell it is necessary that the cell-wall be ruptured, hence in extraction we must first comminute the drug. The cells in some drugs are larger than others, hence some drugs are directed to be powdered more finely than others in various pharmaceutic processes, the aim of the pharmacopœia being to reduce the powder to a fineness sufficient to insure the breaking of every individual cell.

The quantity of cell-wall, as compared to the amount of constituents, is usually many times greater. Thus, for example, in cinchona bark we find that but 5 per cent. of the total amount is alkaloids, and no more than 30 per cent. of the whole is soluble matter. Therefore the primitive method of administering the crude drug in powdered form meant the consumption, by the patient, of a large amount of not merely inert, but indigestible woody tissue, in order to get a small amount of medicament. To obviate the necessity of administering this large quantity of inert matter is the prime object of extraction, and in each case cited above it is accomplished by treating the drug with an appropriate solvent, and the removal, by solution, of the useful principles from the inert insoluble residue.

MACERATION

Maceration is the process of removing the active principles from a drug by allowing the latter to remain in contact with the solvent several days, with frequent agitation.

Roughly stated, maceration consists of reducing the drug to appropriate fineness of powder, placing same in a suitable bottle, pouring on the solvent, which is called *menstruum* in all cases of extraction, and letting the mixture stand for a week, shaking at frequent intervals. The solution is then removed from the inert matter by filtration or straining, and the yield of filtrate increased by subjecting the residue to expression.

The process of maceration is a favorite method of extraction in most European countries. In America maceration is used only where percolation is precluded, and it is, therefore, limited to the few drugs the gummy or viscid character of which would cause a clogging of a percolator. The German Pharmacopœia directs maceration exclusively in the manufacture of tinctures, and even in this country some of the older pharmacists still cling to the process of maceration in the preparation of a large number of tinctures, the advantages they claim for maceration being, first, that the drug need not be powdered as finely as in the case of percolation, second, that the process requires less skill than does percolation, and hence requires less care in manipulation, and, third, that in the process of maceration there is less loss of alcoholic *menstruum* than in making the same preparation by percolation.

The latter is practically the only advantage possessed by maceration over percolation, the other questions not being worthy of attention of the intelligent and educated pharmacist.

In large quantities of a preparation made by maceration, say, gallon lots, the shaking of the bottle containing the drug and *menstruum* is a laborious task, and since the shaking is merely to bring all portions of the solvent in contact with the drug, and as the same result can be obtained by the process of circulatory solution (p. 121), this method can be employed in the maceration of tinctures, the ground drug being placed in a cloth bag and suspended directly below the surface of the solvent.

As mentioned above, the inert residue remaining after maceration (which is called the *marc*) is impregnated with the same strength of tincture, and in filtering off a macerated tincture—say, of *arnica*—the residue on the filter is not wet with alcohol, but with full strength tincture. Hence, it can be seen that the process, even at its best, is wasteful; that the drug is never completely exhausted of its active principles, and the less care used, the more waste there is to the process. The waste is partly obviated by “squeezing the dregs,” as we commonly say; by expressing the residue.

EXPRESSION

This is the process of removing the liquid from a moist mass by the use of pressure, one of the best illustrations of the application of expression being in the removal of the remaining tincture from the *marc*.

Apparatus.—Many forms of presses have been devised, each adapted for some special phase of expression.

All forms of presses, with the exception of forcible straining apparatus and the enterprise screw press, consist of two plates between

which the substance is placed, and which are made to approach each other by means of lever, screw, or some other mechanical power.

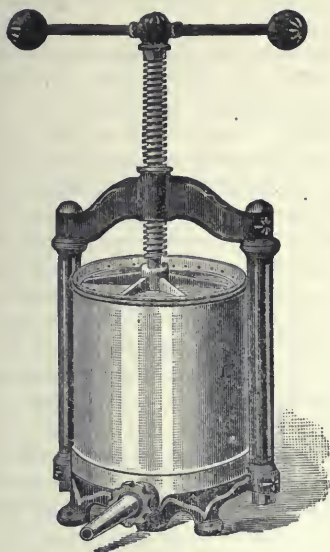


Fig. 147.—Tincture press.

Important in the process of expression is the straining of the expressed liquid through suitable cloth. We, therefore, usually inclose the mass to be expressed in a bag of stout cloth (press cloth) before placing it in the press.

Such press cloth (strong toweling will do) can be used directly for expression, as already explained under *Forcible Straining* (p. 127).

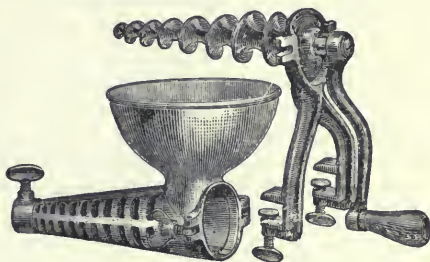


Fig. 148.—Enterprise press (opened).

The *spiral twist press* is based on this principle, but is entirely, useless in pharmacy, hence its bare mention will suffice.

Other forms of presses of little pharmaceutical value, but used in other technical lines, are the *wedge press*, the *roller press*, which

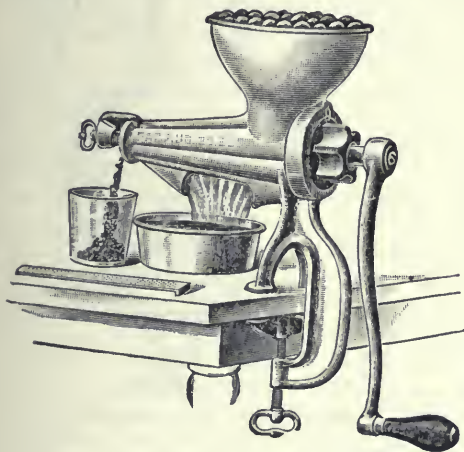


Fig. 149.—Enterprise press.

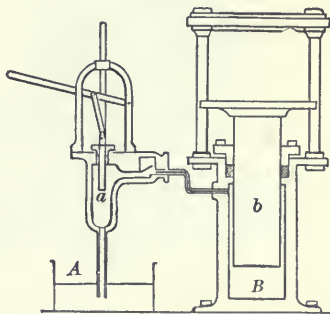


Fig. 150.—Hydraulic press (schematic).

operates on the same principle as the roller clothes-wringer, and is seen in its form of highest perfection in the rollers used for expressing the juice from the sugar-cane, and the *hand-lever press*, in which the upper plate is pressed down upon the lower plate by means of a lever.

In modern pharmacy expression is performed with the *screw press* or *tincture press* (Fig. 147), in which the moist mass is subjected to pressure between two steel plates, the upper one operating on a screw, such as is seen in the ordinary letter-press. A very striking modification of the screw press is the *Enterprise press*, which will best be understood by an examination of Figs. 148 and 149.



Fig. 151.—Hydraulic press.

Of great value and increasing popularity is the *hydraulic press*, the only objection to which is its initial cost. This is constructed on that principle of hydrostatics which asserts that if a liquid be in communicating tubes,—one larger than the other,—any pressure on the surface of the liquid in the smaller tube will be exerted on the surface of the liquid in the larger tube in exact proportion to the relative size of the two tubes. This can be explained more clearly as illustrated in Fig. 150.

Suppose, in this sketch, that the area of the cylinder A is 1 square inch, and that of cylinder B is 10 square inches; then, if we exert a force of 1 pound on piston "a," we will get a force of 10 pounds on piston "b." By using a lever on piston "a" we can so multiply the force that a pressure of 10 pounds on the handle of the lever connected with piston "a" can readily exert the pressure of 1000 pounds on

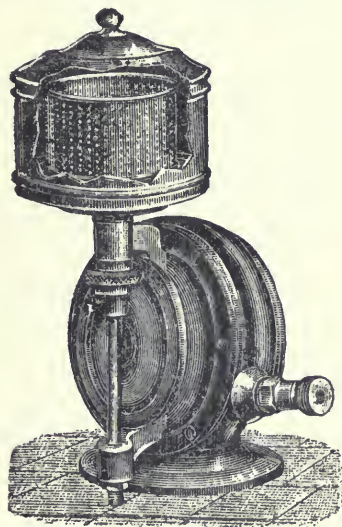


Fig. 152.—Centrifugal extractor.

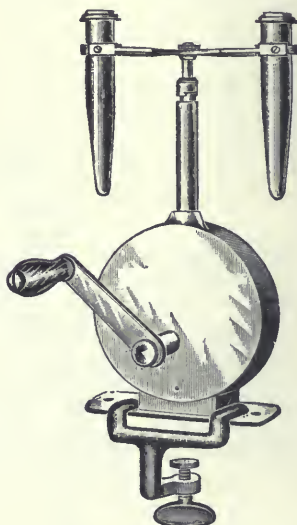


Fig. 153.—Urinary centrifuge.

piston "b"; hence the great value of the hydraulic press, with which comparatively little power can be concentrated into enormous pressure.

An interesting form of removal of liquid from a moist mass without the aid of a press is the operation of the centrifugal machine, in which case the moist mass is placed in a perforated cylinder, which revolves in a large cylinder with velocity great enough to cause practically all traces of the liquid to be thrown out of the inner into the outer

cylinder (Fig. 152). By means of such a centrifugal press 200 or 300 pounds of moist sugar can be centrifuged to almost dryness in the space of five or ten minutes. A modern application of the centrifugal machinery, totally different from expression, is the well-known centrifugal sedimenter, for the precipitation of blood and of urinary sediment (Fig. 153).

PERCOLATION

Percolation is the process of depriving a drug of its soluble constituents by passage of a solvent through the powder contained in a suitable vessel. The solvent in this case is called the *menstruum*, the vessel is called the *percolator*, and the solution of active principles emerging from the percolator is called the *percolate*.

There is no exaggeration in saying that percolation is a distinctive American process. Although the forerunner of percolation—*lixivation*—was used in extracting lye from ashes long before America was discovered; although the first pharmaceutical application was made by Count Real, a Frenchman; to Americans is due the credit of bringing the process to its present stage of perfection. Introduced in the pharmacopœia of 1840 in the preparation of a few tinctures, it has so grown into favor that it is now employed in the manufacture of almost all the tinctures and fluidextracts of the present pharmacopœia, besides being employed in preparing wines, vinegars, oleoresins, and extracts. Foreign countries have not taken kindly to the process, and in the German Pharmacopœia of to-day maceration is largely used in preference. However, percolation is beginning to attract the attention of the Germans, and it was amusing to note, a few years since, in a leading German pharmaceutical journal, a long account of what was called “a new process called percolation.”

Principle of Operation.—Careful study of the definition of percolation will show that in the process advantage is taken of the attraction of gravitation, for it is hardly necessary to say that a liquid poured on top of a powder contained in a suitable utensil will gradually penetrate that powder in a downward direction, and if there is an orifice in the bottom of the vessel, the descending liquid will emerge from the vessel, being attracted by gravity toward the center of the earth. This downward force of gravitation is aided by another force, merely a modification of the former, namely, the weight of the column of liquid above the powder. Mention of the latter is made here merely because the downward force varies according to the height of the column of liquid, whereas the force of gravitation remains unchanged. If this combined action of gravitation was the only force coming into operation, the process would be a simple one. However, there is an opposing force which comes into play during the process of percolation, namely, the upward force of capillarity.

The term *capillarity* comes from the Latin term “*capilla*,” meaning hair, because it was first observed that in every narrow, hair-like tube liquid was drawn up by the cohesive power of the walls of the tube, and, as already explained, to this same force is due the appearance of the meniscus (p. 46). All bodies possessing orifices more or less narrow have this property of drawing up liquids by their own cohesive force, and such is eminently the case with most vegetable

drugs, since, as is mentioned above, they consist of a mass of cells, each cell consisting of an orifice of more or less fineness, surrounded by a cell-wall. In other words, cells can be compared to long sealed tubes, and when the cells are broken by powdering, we have a vast mass of tubes, each possessing capillary attraction. This capillarity explains why a so-called porous substance, for example, a sponge, will absorb so much more water than a non-porous substance, such as sand.

To get back to percolation, besides the force of gravity and its modifications, the pressure of the column of liquid above, forcing the solvent downward through the drug, we have at the same time the capillarity of the cells of the drug attracting the menstruum in the opposite direction. The moment the solution commences to emerge from the vessel, the capillary attraction of the cells tends to keep the liquid within the substance; therefore, acting against the force of gravity. Experience shows that the capillary force of the drug is usually greater than the simple gravitating force of the earth, for unless the gravitating force of the earth is reinforced by the height of the column of liquid above the powder, the liquid ceases to drop from the percolator. As the liquid passes through the drug on its downward course it penetrates the interior of each cell (provided the substance is in fine enough powder) and removes from the same the soluble constituents. Passing from one series it penetrates the next tier of cells, and, taking up more active principle, such solution continues until the solvent is saturated with the soluble principles of the drug. When once a saturated solution, it mechanically passes through the rest of the drug, and finally emerges from the orifice at the bottom of the percolator, providing always that there is a sufficient column of the liquid to force it down. Its passage is followed by a fresh portion of the solvent, which passes through the same cells which it has formerly penetrated, removing the last vestiges of soluble matter, and finally emerging from the percolator also a saturated solution. In this way menstruum is continually poured on until the cells have been so completely exhausted of their soluble constituents that the final portion of the menstruum comes through devoid of color and taste, whereupon the drug is said to be *exhausted*.

The great advantage percolation possesses over maceration is in the fact that, by means of percolation, we are enabled completely to remove every trace of soluble matter from a drug, inasmuch as the saturated solvent does not remain in contact with the drug (if the menstruum column is maintained), but passes from it, being followed by a fresh portion of solvent, which removes the remainder of the soluble principle.

Apparatus.—The utensil in which the drug is packed is called a *percolator*, and consists of a suitable cylindric vessel of metal or glass, provided with an orifice at the bottom. In selecting a percolator those of glass should be chosen when possible, metal percolators having the double disadvantage of being more easily attacked by acid or other substance which the menstruum or drug may contain and also the fact that their opacity prevents watching the operation. Of course, however, there are certain limits to the size of a glass percolator, the writer's experience being that the use of a glass percolator larger than

two gallons is an expensive luxury, inasmuch as such are costly and are quite liable to break. Above two gallons, recourse was had to the galvanized iron percolator or the well tube percolator, in which the container is an earthenware crock.

Shape of Percolators.—The simplest form of percolator is the conic (Fig. 154), consisting of an ordinary chemical funnel. This, however, offers too great a width of surface for the menstruum, which, therefore, has a correspondingly smaller course to travel before emerging from the bottom of the percolator, and, therefore, the familiar tapering percolator came into use (Fig. 155). At the present time the Oldberg cylindric percolator (Fig. 156) is very popular, for by means of it we are able to exhaust a drug far more thoroughly than in either a tapering or a conic percolator, from the simple fact that each portion of menstruum has to travel through a larger amount of the drug before emerging from the percolator. A



Fig. 154.—Conic percolator.



Fig. 155.—Tapering percolator.



Fig. 156.—Oldberg percolator.



Fig. 157.—
Soxhlet ex-
tracting ap-
paratus.

variety of percolator used exclusively in chemistry is the Soxhlet condenser (Fig. 157).

The saturated solution, when emerging from the percolator, is called the *percolate*, and, of course, this must be collected in an appropriate vessel. For this purpose a wide-mouthed bottle, the receiving bottle, is usually employed. Elaborate forms of receiving bottles are on the market in which the graduations are engraved upon the glass. These, while presenting an elegant appearance, are scarcely necessary, as the pharmacist has many wide-mouthed bottles at his disposal, and he can usually graduate same by pouring in definite quantities of liquid from an accurate graduate, and marking amount by cutting with a file or on a strip of paper pasted along the length of the bottle (marking same with ink). The percolator must be supported on an appropriate stand, and for this purpose nothing can be better than the retort stand pictured in Fig. 77, it being always understood, however, that the glass percolator must never

come in contact with the iron ring, being protected from the same by covering the ring with paper or cloth (Fig. 158).

When large amounts of percolation are being carried on, special percolating stands have been devised; such, for example, is the Shinn percolating closet and the Remington percolating shelf ("Am. Jour. Pharm.," 1877, p. 589).

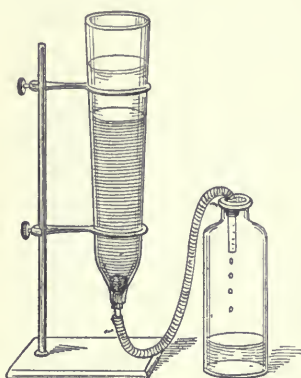


Fig. 158.—Correct percolation.

Operation.—The process of percolation may be divided under the following headings: Comminution of the drug; moistening the same; packing in percolator (with maceration either before or after packing); pouring on the menstruum and collecting percolate until exhausted.

Comminution.—Before percolating the drug it is essential that it be reduced to particles of more or less fineness, for the same reason that we comminute drugs before solution. As mentioned above, in percolation it is essential that the drug be ground sufficiently fine to rupture all the cells of which it is composed, and this degree of fineness is dependent upon the structure of the drug, those drugs having small cells calling for a finer degree of comminution than those in which the cells are large. Thus, *nux vomica* must be powdered very finely, whereas in gentian the cells are large and a coarse powder can be utilized. This explains why the pharmacopœia directs various degrees of fineness for the powders used in percolation. Another point regarding comminution of the drug is the rapidity or completeness of exhaustion. Thus, in percolating fluidextracts it is usually desirable to exhaust the drug with less menstruum than in preparing tinctures from the same drug, and in order to do this the drug used in percolating a fluidextract is generally directed to be in finer powder than that used in making the tincture. The powdering of the drug is accomplished by those methods of comminution described in Chapter VI. best serving the particular purpose, the main requirement being that the drug used in percolation be of uniform fineness.

Moistening of the Drug.—Before packing the drug in the percolator it is directed to be moistened with menstruum, this being done because, when the drug comes in contact with the menstruum, the compressed dried cells are swollen to their normal size, and if this swelling occurred within the percolator, it would cause sufficient expansion to cause a stoppage of the operation.

This moistening is accomplished by placing the drug in a suitable container, preferably of porcelain or stoneware, adding the required quantity of menstruum, and mixing same thoroughly with the drug by stirring with a wooden paddle. After being moistened it should be allowed to remain for half an hour in order to complete the swelling, thereupon the pharmacopœia directs the moist drug to be passed through a coarse sieve, thus making the product into a coarser granular powder, ready for packing.

Packing the Drug.—To receive this granular powder the clean and

absolutely dry percolator is prepared by placing in its neck a plug of absorbent cotton, followed by a layer of clean dry sand. This latter precaution is directed by the pharmacopœia, both for the purpose of filtration and also to hold the plug of cotton in place. Many careful pharmacists, however, omit the sand as an unnecessary detail. Upon this sand the granulated powder is poured in comparatively small quantities, the usual rule being to place in the percolator about one-fourth at a time, and each fourth is carefully and firmly packed before the next portion is added. The packing is accomplished by means of a wooden plunger, and for this purpose a piece of wood will suffice, there being nothing better than a wooden potato-masher. The packing of the drug is a matter of skill, and in order to do it successfully it is necessary to know the character of the drug and the menstruum. Drugs in finer powder are not, as a rule, packed so tightly as those of coarser powders, but the supreme test of the correct packing is the rapidity of the flow of the percolate. If the drug is packed too tightly, the percolate drops too slowly, and if too loosely, the percolate runs so rapidly as not fairly to exhaust the drug; therefore, packing must be put down as chiefly a matter of experience. The nature of the menstruum also has an influence on the packing of the percolator. If the menstruum is pure alcohol, the drug can generally be packed more tightly than if it consists of water, and if glycerin is present in the menstruum, care should be taken not to pack the drug too tightly. It is hardly necessary to say that in packing the drug the pressure should be equal on all sides of the powder. If the drug on one side of the percolator is packed more loosely than on the other, it stands to reason that the menstruum will choose the easiest possible way and will run down the looser side, and, therefore, the drug will be incompletely exhausted.

All the drug being packed in the percolator, there is carefully placed above it a sheet of filter-paper which is held in place by a glass weight, any unused glass stopper answering the purpose. The object of the paper is to prevent the disturbance of the powder when the menstruum is poured in, the force of the fall of the liquid being apt to cause the rising of the powder to the surface of the menstruum.

Instead of filter-paper, the writer found very convenient disks of ordinary clean Manila card-board, made from one of the many rejected paste-board boxes found in every drug-store. These disks, perforated by the point of a file, when just the size to fit snugly above the drug within the percolator, required no weight to keep them down.

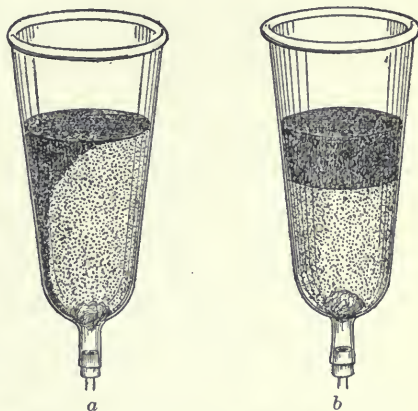


Fig. 159.—*a*, Improper packing; *b*, proper packing.

Whether the drug has been packed evenly on all sides is shown by the descent of the first menstruum, which should go down in a perfectly even ring, as shown in Fig. 159, *b*.

After the drug is packed in the percolator and enough menstruum has been poured in fully to penetrate the drug and to begin dropping from the percolator, the lower orifice is closed and the drug is directed by the pharmacopœia to macerate within the percolator for a period ranging from twelve to forty-eight hours. Many prefer to macerate the drug for a period of twenty-four to forty-eight hours before packing in a percolator, as in this case the maceration can be conducted in an absolutely closed vessel or a well-stoppered bottle.

Adding Menstruum.—The menstruum is, as mentioned above, the solvent used for extracting the active principles of the drug. The various menstrua directed by the pharmacopœia for different drugs have been carefully selected with a view to using the particular solvent best adapted easily and speedily to remove the active principles of the drug under consideration. In some cases, however, the pharmacopœial committee has been guided by practical commercial considerations, the rule being to choose as menstruum the cheapest solvent that will fully answer the purpose. Understanding this fact, the pharmacist should carefully follow the pharmacopœial instructions, appreciating that when the United States Pharmacopœia directs strong alcohol, it is because a weaker alcoholic menstruum will not answer the purpose.

Usually the menstrua used in percolation are of various mixtures of alcohol and water; occasionally glycerin is directed to be added to the menstruum, this usually in cases of drugs containing large quantities of tannin. In the same way ammonia water is added to the menstruum in preparations of licorice, and ammonia or other alkalis for drugs containing pectin, while acids are used in the menstrua for drugs containing volatile alkaloids, such as conium, ergot, etc.

Not all percolates are made with alcoholic menstrua; for example, some official infusions are made by percolation of the drug with water—infusion of cinchona (U. S. P. 1890) with water plus aromatic sulphuric acid, and infusion of wild-cherry (U. S. P. VIII.) with cold water.

Again, stronger white wine is used as a menstruum in the preparation of percolated wines of the pharmacopœia of 1890. Dilute acetic acid is used in those vinegars made by percolation, while oleoresins are made by percolating the drugs with acetone.

The Percolation.—The proper menstruum is poured upon the filter-paper above the powdered drug in the percolator, care being taken to pour it so as not to cause the floating of the paper. As the menstruum passes down through the drug, more is added to take its place, it being an unvaried rule that there should always be maintained a layer of menstruum above the surface of the drug, for, if the drug is exposed to the air, evaporation takes place from its surface, and fissures occur through which the menstruum added afterward will run, instead of through the entire surface of the drug. In order to keep a sufficient quantity of menstruum upon the surface of the drug, recourse is had to continuous percolation by inverting the menstruum bottle within the percolator in the same manner as suggested in con-

tinuous filtration (Fig. 160). The menstruum poured in and passing through the drug should emerge from the percolator slowly and steadily, the rapidity being dependent entirely upon the quantity of the drug being manipulated; thus the last pharmacopœia directs a flow of two to five drops a minute in percolating 1000 grammes of drug for fluidextract, while for tinctures the flow is to be eight to fifteen drops a minute. It is needless to say, however, that this standard would be absurd if we were percolating 200 pounds of a drug and expecting, say, 100 gallons of the percolate.

In most operations in retail pharmacy, however, in which we deal with 200 to 1000 Cc. of menstruum, the speed given above may be maintained, and a skilful operator aims to so pack his drug as regularly to attain this speed, and after some practice usually learns the art. In order, however, to insure the speed of percolation directed, the pharmacopœia directs that the lower orifice of the percolator be closed with a cork through the perforation of which passes a glass tube which is fitted with a rubber tube, at least as long as the percolator and terminating with a second glass tube bent in the form of a "V." When the rubber tube is raised so that the outlet is above the surface of the menstruum in the percolator, it follows that no liquid will run out, whereas if it is lowered to its greatest depth, the liquid will run out very rapidly, and, by raising or lowering the tube to its greatest height or greatest depth, the rapidity of the percolation can be regulated to a nicety. If it is desired to close the tube completely, a pinch-cock (p. 99) can be used, although if the tube is of sufficient length, this is scarcely necessary.

In practice, the rubber tubing is not so convenient as it seems at first blush, as the several joints of the cork, the glass tube, and the rubber tube fittings are apt to leak unless very carefully prepared, making an unsightly and dirty process. If, therefore, the percolating tube is used, care should always be taken *before percolation* to see that the cork and rubber tubes do not leak by pouring water into the empty percolator, raising the tube to its greatest height, and allowing it to stand in that way for some time.

Many pharmacists omit the use of the rubber tube, substituting therefore an ordinary cork with notches in the side, which do not extend through the entire length. If the percolation is desired to be stopped, the cork is pushed up into the neck beyond the notch, when an absolutely water-tight fitting occurs. When the flow is desired, the cork is loosened so as to allow a part of the notch to be beyond the neck of the percolator, leaving a tiny orifice through which the liquid can be dropped. This scheme is usually satisfactory,

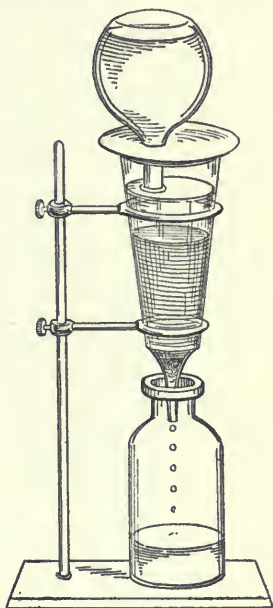


Fig. 160.—Continuous percolation.

although the tightening and loosening of the cork during the process of percolation are apt to be untidy. Far better, therefore, is the use of a metal sprinkler top, such as is used for tooth-wash bottles.

Finishing Percolation.—By making preparations by percolation it is generally intended that the percolation be continued until the drug is exhausted, the word meaning in this case the removal from the drug of all its soluble constituents. There are several ways of proving the practical exhaustion of a drug, for, in the operation, it is usually unnecessary to carry on percolation to the extreme point of removing every particle of soluble matter. If the drug is a bitter one, we generally say it is exhausted when the percolate comes through no longer bitter, even though it may be still impregnated with some of the coloring-matter residing in the drug, the latter being more tenacious and persistent of removal. In some cases the bitterness is so great that a large amount of menstruum will be necessary before every trace of bitterness is removed. An example of this is quassia, in which case we generally consider the drug exhausted when the percolate is almost free from bitterness.

Tannin-bearing drugs are usually percolated until the percolate is free from astringency, whereas in odorous drugs the absence of odor from the percolate announces exhaustion.

The residue left after percolation or maceration is called the *marc*, and in both percolation and maceration it retains a considerable amount of moisture. As mentioned under Maceration, in the latter case the moist liquid is the finished full strength tincture, and this marc should always be expressed, else the yield of tincture will be lessened. In percolation the marc is moist with the menstruum, hence there is no loss so far as finished tincture is concerned, but the loss of menstruum is excessive. It, therefore, behooves the economic pharmacist to recover the alcohol from this marc, which can be done by the process of steam distillation. During the writer's experience in practical pharmacy it was his custom to keep on hand a number of half-gallon wide-mouthed candy jars, securely corked, and in these he threw the successive batches of marc at the end of every percolation. When sufficient quantity had accumulated in these bottles, his steam distillation apparatus was fitted up, the tube from the boiler fitted into the candy bottle, and steam passed through and the vapors collected. The yield of alcoholic distillate more than paid for the fuel and time employed.

This process is found more convenient and efficacious than the ordinary method of pouring water on the drug, and thus forcing the menstruum out, as in the latter case the addition of water to the starchy matter remaining in the marc is apt to cause a swelling of the drug and subsequent clogging of the percolator.

Modified Percolators.—Under Distillation and Comminution we found that ingenuity had devised many special apparatus for carrying on both operations, and the same also holds good in regard to percolation.

Chief among these improved forms of percolators must be mentioned the Squibb's well tube percolator (Fig. 161), which is primarily designed for the percolation of large quantities of drug. As mentioned above, under the consideration of the percolator itself, glass or

earthenware percolators larger than two gallons are inconvenient to handle. Therefore, in large manufacturing establishments recourse is had to either huge metal percolators or to percolators made of wood. Metal percolators are not advisable, for acids from the drug or menstruum may attack the metal and render the product liable to contamination by metallic contact, while wooden percolators, even if unobjectionable from the standpoint of contamination, becoming saturated from contact with an alcoholic solution of a certain drug, can be used for the percolation of that drug alone, and, therefore, in their use a separate percolator must be provided for each drug desired to be percolated. The chief cause of the frailty of glass or earthenware percolators is their funnel form, which also increases their cost. Earthenware crocks with flat bottoms can be obtained of almost any size and for prices comparatively low, and, possessing a firm foundation, are very durable; hence the use of same in percolation was suggested to the fertile mind of Dr. Squibb, whose experiments brought forth the well tube percolator, which consists of an earthenware crock with the inside bottom fitted with a sheet of felt for filtering purposes, and the drug packed therein around a large glass tube. If, upon the drug so packed, menstruum is poured, it percolates through to the felt, then rises in the interior of the central glass tube to

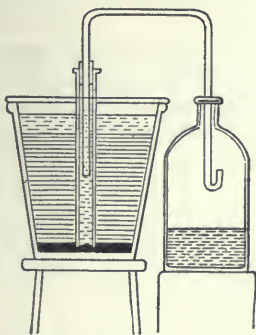


Fig. 161.—Squibb's well tube percolator.

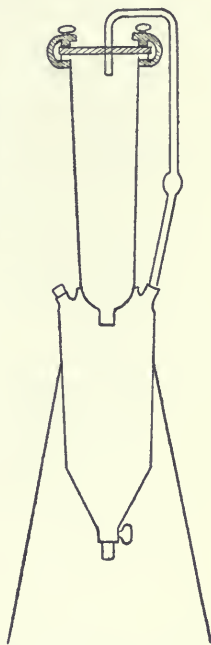


Fig. 162.—Economic percolator.

practically the same height as it is on the outside, following the well-known law of water seeking its own level.

Now, if in this tube is placed a syphon, the liquid within the well tube can be drawn off, and if a sufficient quantity of menstruum is kept on drug outside the well tube, a constant level can be maintained in the well tube ready to be drawn off through the syphon. In order to maintain a constant flow from the syphon its lower end must be bent up as shown in cut, making the orifice at least three-quarters of an inch higher than the orifice of the short arm. If this precaution is not taken, the liquid in the well tube is syphoned off more rapidly than it percolates, emptying the syphon immediately (Fig. 161).

Among the other forms of modified percolators are the various pressure percolators, such as Rosewater's and Berry's, both of these being based on the principle of the Real percolating press. With these, drugs can be percolated very rapidly and quite thoroughly.

Volatile Liquid Percolators.—In the manufacture of oleoresins by percolation with ether care must be taken to avoid undue evaporation of the volatile solvent, and in this case a special variety of percolator (Fig. 162) has been suggested. A similar volatile liquid percolator can be made with any ordinary percolator by fitting the lower orifice of the percolator with a cork, through which passes a glass tube, which in turn passes through another cork tightly fitted into the receiving bottle (Chap. XV.). Then there must be an exit for the air from the receiving bottle, hence the receiving bottle must have two orifices, one provided with a glass tube connected by a rubber tube to the cork, tightly closing the top of the percolator; which cork, for convenience sake, is usually provided with a second orifice, through which passes a funnel for the admission of the solvent. In some works the use of the rubber tube connecting the receiving bottle with the top of the percolator is objected to on the grounds that the vapors of ether will attack the rubber of this tube, hence it is recommended that the outlet tube of the receiver be drawn to a fine, tapering point, to reduce loss of evaporation to the menstruum, a similar tapering tube being fitted in the cork closing the top of the percolator. The author's experience, however, has shown that the danger of destroying the rubber is scarcely worth considering, and not only by means of such rubber tubing is the loss by evaporation reduced to the minimum, but a steady flow of air from the receiving bottle to the top of the percolator ensues.

A modification of a volatile liquid percolator was used by the writer in a long series of experiments in ordinary percolation, from which he drew the conclusion that it would be highly profitable to use such a volatile liquid percolator for all cases of percolation. ("Proceedings A. Ph. A.," vol. xl, p. 169.)

PART II

INTRODUCTION

GALENIC PHARMACEUTIC PREPARATIONS

HAVING considered all the processes used in pharmacy, we are now ready to study the application of these in the manufacture of pharmaceutical preparations.

For convenience in teaching we group pharmaceuticals into two classes—*galenic preparations* and *chemical preparations*. Those preparations in the manufacture of which no chemical action is involved are called galenic, after Galen, one of the ancient medical authorities, while chemical preparations are those in which a chemical change is produced during process of manufacture.

Already, in discussing Solution (p. 119), attention was called to the fallacy of the grouping into simple and chemical solution, from the strictly philosophic standpoint; yet, at the same time, the convenience of such grouping from the pharmaceutical point of view is readily admitted.

The same reasoning applies to a certain extent to the grouping of pharmaceuticals into galenic and chemical preparations, for many so-called chemical preparations can be made equally well by the retail pharmacist by purely galenic means. Thus, while the manufacture of syrup of hydriodic acid of the pharmacopœia of 1890 involved a chemical action, the recipe of the present pharmacopœia consists in merely mixing diluted hydriodic acid with water and syrup. It is true that in making diluted hydriodic acid (U. S. P. VIII.) the same chemical action occurs that took place in making syrup of hydriodic acid (U. S. P. 1890), but this does not apply as far as the present syrup is concerned, it being just as much a galenic as is syrup of citric acid.

As above mentioned, the classification of pharmaceuticals into galenic and chemical preparations, as far as this book is concerned, is a matter of convenience.

It would be manifestly absurd to discuss the manufacture of a preparation of which the most prominent feature is a chemical reaction, before considering the subject of chemistry as outlined in Part III.; hence in all such preparations the discussion of their manufacture will be deferred until Part III. is reached, and the only mention of them in this division will be the grouping of them under the heading Chemical Preparations in the list of officials found at the beginning of the discussion of each group of official pharmaceuticals.

The groups of official pharmaceuticals are given below in a tabulated form:

LIQUID PREPARATIONS

- I. Solutions of volatile substances:
 - Aqueous.....*Waters.*
 - Alcoholic.....*Spirits.*
- II. Solutions of non-volatile substances:
 - (a) Made by a simple solution:
 - Aqueous.....*Solutions.*
 - Aqueous (viscid).....*Mucilages.*
 - Aqueous (saccharine).....*Syrups.*
 - Mellifluous.....*Honeys.*
 - Alcoholic.....Some *tinctures*, as tincture of ferric chloride.
 - Alcoholic (saccharine).....*Elixirs.*
 - Glycerinic.....*Glycerites.*
 - Ethereal.....*Collodions.*
 - Oleaginous.....*Oleates.*
 - (b) Made by maceration or percolation:
 - Aqueous.....*Infusions and Decoctions.*
 - Alcoholic.....*Tinctures and Fluidextracts.*
 - Vinous.....*Wines.*
 - Ethereal.....*Oleoresins.*
 - Acetous.....*Vinegars.*
- III. Liquids containing undissolved matter:
 - (a) For internal use:
 - Aqueous, with neither oil nor resin.....*Mixtures.*
 - Aqueous, with oil or resin.....*Emulsions.*
 - (b) For external use:
 - Oleaginous.....*Liniments.*

SOLID PREPARATIONS

- I. Made by maceration or percolation:
 - By evaporation.....*Extracts* (*Abstracts*, U. S. P. 1880).
 - By precipitation.....*Resins.*
- II. Without maceration or percolation:
 - (a) For administration by mouth in undivided portions:
 - Pulverized.....*Powders.*
 - Semisolid.....*Masses.*
 - Semisolid (saccharine).....*Confections.*
 - In divided portions:
 - Globular doses.....*Pills.*
 - Disk-like doses.....*Troches.*
 - (b) For administration by rectum, etc.....*Suppositories.*

(c) For external administration:

Greasy masses—

Applied as a plaster....*Cerates.*

Applied by inunction...*Ointments.*

Moist masses.....*Cataplasms.*

Sticky masses.....*Plasters.*

Spread on or absorbed by
paper.....*Papers.*

CHAPTER XI

WATERS AND SPIRITS

WATERS

WATERS are *aqueous solutions of volatile substances*, not like spirits, which are alcoholic solutions of volatile substances; not like solutions, which are aqueous solutions of non-volatile substances.

Again, the volatile substance may be solid, liquid, or gaseous. An example of an official water containing a volatile solid is found in camphor water; among those containing volatile liquids may be cited any of the official waters made from a liquid oil.

The following table gives a list of official waters grouped according to process of manufacture:

TABLE OF OFFICIAL WATERS

Process.	Galenic Preparations.	Chemical Preparations.
Simple solution or dilution.....	<i>Ammonia water.</i> <i>Bitter almond water.</i> <i>Creosote water.</i> <i>Chloroform water.</i> <i>Orange flower water.</i> <i>Rose water.</i>	
Gaseous solution.....		<i>Stronger ammonia water.</i> <i>Chlorine water</i> (U. S. P. 1890).
Chemical action.....		<i>Solution of hydrogen dioxide.</i>
Filtration through absorbent powder.....	<i>Anise water.</i> <i>Camphor water.</i> <i>Cinnamon water.</i> <i>Fennel water.</i> <i>Peppermint water.</i> <i>Spearmint water.</i>	
Distillation	<i>Distilled water.</i> <i>Hamamelis water.</i> <i>Stronger orange flower water.</i> <i>Stronger rose water.</i>	

The process of preparing by *simple solution* or *dilution* consists in taking the constituent and adding and agitating same with a sufficient quantity of water. With those prepared by *gaseous solution* the gas is generated in an appropriate apparatus, and, after being washed, is passed into the cylinder containing the water, as explained

on p. 123, usually the passage of the gas being continued until the solution is saturated with gas. This explains the discrepancy in the strength of stronger ammonia water (28 per cent. ammonia gas) as compared with chlorine water (U. S. P. 1890) (0.4 of 1 per cent. chlorine gas), these widely varying strengths representing the saturated solutions of the respective gases in water.

Filtration through Absorbent Powder.—This process is employed to obtain a saturated solution of volatile oil, the only exception being the manufacture of camphor water, which see. Volatile oils are very sparingly soluble in water, and, in the attempt to secure a more thorough solution, the oil is separated into a finely divided form by treating with an absorbent powder, and when thoroughly blended with this absorbent, the water is gradually added, and in this way the finely divided liquid is intimately combined with all portions of the water. The absorbent powders employed for this purpose in former pharmacopœias were magnesium carbonate and precipitated calcium phosphate. Waters prepared with either one of these powders are unsatisfactory. Magnesium carbonate dissolves in water in appreciable amounts, and an aromatic water prepared in this way will very frequently precipitate chemicals one desires to dissolve therein. Calcium phosphate was chosen by the pharmacopœia of 1890 by reason of its supposed insolubility; but investigation showed that while calcium phosphate is not so soluble as magnesium carbonate, the minute traces which do dissolve cause more mischief than does the magnesium carbonate; the calcium phosphate acting as pabulum for the spores of microscopic plants which may lodge in the water, forming flocculent, slimy masses, due to the growth of these spores into plants.

For this reason, in the last revision, purified talc was chosen as the absorbent powder.

Distillation.—This process was the first way in which aromatic waters were prepared, and yields the finest product. The process of distillation has been fully described in Chapter V., and here need only be mentioned the modifications of the process adapted for the product under consideration.

The substances from which the volatile principles are to be obtained are suspended in a boiler or a still, and are never permitted to lie on the surface of the boiler next to the flame, for in such a case the heat produces a decomposition, and the product will be ruined through presence of empyreumatic odors. Into the still a requisite quantity of water is poured and is heated on a slow fire, and, as the vapors rise from the boiler, they carry with them the aromatic principles found in the plant. If enough of the substance is used, a considerable excess of the oil may be found floating on the surface of the distillate. In fact, most distilled waters are made on a large scale as the side product in the distillation of volatile oils (see p. 816).

Distilled rose water and orange flower water of commerce are termed simple distilled, double distilled, triple distilled, or quadruple distilled, according to strength.

A double distilled water is prepared by mixing 1 part of flowers with, say, $2\frac{1}{2}$ parts of water, and collecting 2 parts of distillate; triple distilled is made by distilling off one part of distillate from $1\frac{1}{2}$ parts

of flowers; while quadruple distilled is made by securing 1 part of distillate from 1 part of flowers; hence the triple distilled is stronger than the quadruple distilled. In some cases, in preparing quadruple distilled water, another 1 part of flowers is mixed with sufficient double distilled water, and 2 parts distillate obtained. This process of redistilling is called *cohobation*.

In most of the modern perfume factories the distillation over a naked fire has been supplanted by distilling in steam. In this case steam is passed into an appropriate iron vessel containing the aromatic substance, and in passing through it carries the odor along with it.

The preservation of distilled waters is not an easy matter by reason of the fact that such solutions afford a nidus for the growth of microscopic plants.

It has been observed that the growth of these plants is furthered by the corking of the containers, and in the warehouses of the large aromatic water distillers the waters are kept in carboys, which are closed with a pledget of cotton instead of being corked.

The term "aromatic waters" has reference to that class of waters which are made from odorous principles, and are used chiefly as perfumes and flavorings. This includes all the official waters except the ammonia waters, creosote water, chloroform water, hamamelis water, solution of hydrogen dioxide, and chlorine water (U. S. P. 1890).

Two methods of manufacture of aromatic waters not given official sanction are by *solution in hot water* and by *percolation through cotton*.

Hot-water Process.—This consists in agitating volatile substances with *hot water*, a process recommended by several pharmaceutic writers. The process, however, is not official, and the pharmacopœia has been wise in omitting the same, for the reason that the use of hot water occasions considerable loss through the evaporation of the volatile substance. Again, it may be cited that the use of hot water does not produce more saturated products; experiments have shown that when the waters so prepared are brought to the normal temperature and filtered until perfectly clear, their strength is no greater than those made by other processes. (See "Proc. A. Ph. A.," vol. xlv, p. 234.) As a possible advantage of the use of hot water it has been said that the process afforded more rapid solution than any other process. But, admitting this as true, it is more than counterbalanced by the inconvenience of agitating a hot bottle with the possible blowing out of the cork, to say nothing of the danger of fracturing the glass container.

Percolation through Cotton.—The pharmacopœia of 1880 adopted the plan suggested by W. S. Thompson, of subdividing the oil by pouring it on absorbent cotton and picking the cotton into shreds. In this way a small quantity of oil can be distributed thoroughly over a considerable amount of cotton, and thus become intimately subdivided, strands of cotton acting as a capillary and drawing into the lumen a minute trace of the oil. The process yields a very good and permanent product, but was abandoned in the revision of 1890 by reason of the untidiness of the operation, the pulling of the cotton usually being accomplished by hand. A modification of this process, the principle of which is the same, is that of dropping the oil drop by

drop on a large sheet of filter-paper, and finally tearing the paper into shreds and digesting it in water, but this process is scarcely more advantageous than is the picking of cotton. The cotton on which the oil has been divided is then placed in a percolator, the neck of which has been plugged with a piece of pure absorbent cotton, and the cotton, imbued with oil, is then packed down firmly, and the water is made to percolate through the same. It must be said, in passing, however, that this process does not yield so strong a product as does the filtration through absorbent powder. And, in truth, none of the processes yields a more superior product in any way to the water prepared by pouring the oil into cold water and agitating from time to time during a period of twenty-four hours. Water so treated is separated from the undissolved oil by passing through a well-wetted filter.

It will be noted that the present pharmacopœia gives permission to prepare aromatic waters, either by the filter-paper process, by hot solution, or by distillation under the special heading—*Aquæ*—which is reproduced below.

Special Notes on Official Waters.—Before discussing the several official waters, a few words explanatory of the order of presentation of these and other galenic preparations may not come amiss. Having already given the general methods of manufacture and a table of the official representatives of this class of preparations, at this place reference need only be made to the individual peculiarities of each of the several preparations, which will be presented in alphabetic order, except in cases where other arrangements are advisable. The official recipe for many of the same class of galenics is identical, save the constituents, and, therefore, in order to avoid “vain repetition,” the pharmacopœial recipes will be printed in full only in one case, and under the other preparations made by the identical process will be the reference to the typical recipe, with a note as to the deviations in making the preparation under consideration. To be specific, in the class of aromatic waters the six official waters—*anise*, *camphor*, *cinnamon*, *fennel*, *peppermint*, and *spearmint*—are made by exactly similar process, filtration through the absorbent powder (purified *talc*), the only difference being the employment of the specific oil (or alcoholic solution in one case) needed to make the particular flavor desired. Hence the exact pharmacopœial recipe will be given in case of *anise water*, and in discussing the other five waters reference will be made under “recipe” to the *anise water* recipe, with such changes as are necessary to produce that special official. If no recipe is prescribed by the pharmacopœia, mention of the fact will be given.

Again, as already mentioned, Part II. is not the proper place to discuss the chemistry of manufacture of such official preparations as are more chemical than pharmaceutical; hence in such cases discussion of manufacture will be left for Part III., and the fact so noted.

Lastly, these arrangements will obtain as far as possible in all the different classes of preparations discussed in Part II. Particularly will the “general recipe” idea be applied in the case of *tinctures* and *fluidextracts*, where scores of each class of preparations are made by a process identical, save in choice of *menstruum* and fineness of powder.

AQUÆ. Waters.

Recipe.—The Medicated Waters, when prepared from volatile oils, are intended to be, as nearly as practicable, saturated solutions, which must be clear and free from solid impurities. In the processes which follow, the solution of the volatile oils is facilitated by the use of purified talc; but the solution may, if preferred, be aided by replacing the purified talc by pulped or shredded filter-paper; waters may also be made by the addition of volatile oils to hot water and separation of the excess of the former, or by the distillation of the drug or volatile oil with water, if by either of these methods the finished product corresponds in all respects with official requirements.

Remarks.—The above statement gives pharmacopœial sanction to the manufacture of aromatic waters by the several processes there enumerated.

AQUA AMMONIÆ and AQUA AMMONIÆ FORTIOR.

Both are solutions of the chemical, ammonia, and will be described on p. 484.

AQUA AMYGDALÆ AMARÆ. Bitter Almond Water.

Recipe.—Oil of Bitter Almond, one cubic centimeter..... 1 Cc.
 Distilled Water, nine hundred and ninety-nine cubic
 centimeters..... 999 Cc.
 To make one thousand cubic centimeters.. 1000 Cc.

Dissolve the Oil in the Distilled Water by agitation, and filter through a well-wetted filter.

Remarks.—Note that the strength of this water is 0.1 of 1 per cent.
Dose.—4 Cc. (1 fluidrachm).

AQUA ANISI. Anise Water.

Recipe.—Oil of Anise, two cubic centimeters..... 2 Cc.
 Purified Talc, fifteen grammes..... 15 Gm.
 Distilled Water, a sufficient quantity,
 To make one thousand cubic centimeters.. 1000 Cc.

Triturate the Oil of Anise with the Purified Talc, add the Distilled Water gradually with continued trituration, filter, and pass the filtrate through the filter repeatedly until the Anise Water is perfectly clear.

Remarks.—Anise water is made by trituration of 2 Cc. of oil with 15 Gm. of purified talc and enough water to make 1000 Cc. of the finished product.

From this data it is frequently stated that the percentage of oil in anise water is 0.2 of 1 per cent. This, however, is incorrect, inasmuch as scarcely more than half of the oil is dissolved by the water, the rest remaining in the talcum left on the filter.

Dose.—16 Cc. (4 fluidrachms).

AQUA AURANTII FLORUM. Orange Flower Water.

Recipe.—Stronger Orange Flower Water,
 Distilled Water, each, one volume.

Mix them immediately before use.

Remarks.—This water is made by the process of dilution. A radical change was made in both “rose water” and “orange flower water” in the revision of 1890.

What were known by these titles in the former pharmacopœias—the so-called triple distilled imported waters—are now official as the “stronger waters,” while the terms rose water and orange flower water are applied to waters diluted to 50 per cent. by the addition of distilled water.

An ancient synonym for orange flower water which is sometimes employed in prescription writing is *aqua naphæ*.

Dose.—16 Cc. (4 fluidrachms).

AQUA AURANTII FLORUM FORTIOR. Stronger Orange Flower Water.

Water saturated with the volatile oil of fresh orange flowers obtained as a by-product in the distillation of the oil of orange flowers. It should be kept in bottles loosely stoppered with a pledget of purified cotton, and in a dark place.

Stronger Orange Flower Water should be neutral to litmus-paper, and have a strong odor of fresh orange flowers.

It should be colorless and clear, or only faintly opalescent, not mucilaginous, and should give no reaction with hydrogen sulphide T.S. or ammonium sulphide T.S. (absence of *metallic impurities*).

Manufacture.—No recipe is given by the pharmacopœia, but this water is made by cohobation, as already described on p. 169, the commercial article being “triple distilled.”

Remarks.—The metallic impurity test given above is intended to prevent use of a water that has been kept too long in the copper containers (usually tin lined) in which the commercial article is usually shipped.

Medical Properties.—Flavor and mild sedative.

Dose.—8 Cc. (2 fluidrachms).

AQUA CAMPHORÆ. Camphor Water.

Recipe.—Like anise water, except that in making 1000 Cc. the talc is triturated with a solution of 8 Gm. of Camphor in 8 Cc. of alcohol.

Remarks.—Camphor water is the only official water made by trituration with talc in which the base is not a volatile oil. In making it the camphor is dissolved in alcohol and the alcoholic solution is triturated with purified talc, as if it were a volatile oil.

Dose.—8 Cc. (2 fluidrachms).

AQUA CHLORI (U. S. P. 1890). Chlorine Water.

Replaced in U. S. P. VIII. by *Liquor Chlori Compositus* (p. 391).

The process of manufacture of this will be found on p. 389, as this is not the proper time to consider the chemical action. Chlorine water is a preparation which should be made freshly each time it is dispensed, and hence in the last revision a simpler extemporaneous process leading to a similar preparation was introduced and the name changed to *compound solution of chlorine*.

AQUA CHLOROFORMI. Chloroform Water.

Recipe.—Chloroform,
Distilled Water, each, a sufficient quantity.

Add enough Chloroform to a convenient quantity of Distilled Water, contained in a dark amber-colored bottle, to maintain a slight excess of the former after the contents have been repeatedly and thoroughly agitated.

When Chloroform Water is required for use, pour off the needed quantity of the solution, refill the bottle with Distilled Water, and saturate it by thorough agitation, taking care that there be always an excess of Chloroform present.

Remarks.—The above recipe is a modification of the process of simple solution, the finished product being kept over a layer of chloroform. This is done in order that the finished product remain a saturated solution, experience proving that a filtered chloroform water soon loses all its volatile active principle, leaving nothing but ordinary water. The saturated solution contains about $\frac{1}{2}$ of 1 per cent. of chloroform.

Dose.—16 Cc. (4 fluidrachms).

AQUA CINNAMOMI. Cinnamon Water.

Recipe.—1000 Cc. water is made exactly as is anise water, except that 2 Cc. oil of cinnamon is employed instead of the oil of anise.

Remarks.—Cinnamon water belongs to the same class as anise water, made by the same method, and the remarks applied to the strength of anise water are equally forcible in the case of the water under consideration. It must be borne in mind that the oil of cinnamon from which the water is made is very irritating, and often in the dilution found in cinnamon water the taste is decidedly biting. For this reason the pharmacopœia directs, when making chalk mixture, that the cinnamon water be used diluted with a quantity of water, as explained on p. 265.

Dose.—16 Cc. (4 fluidrachms).

AQUA CREOSOTI. Creosote Water.

Recipe.—Made like bitter almond water, by simple solution, except that 10 Cc. of creosote is mixed with 990 Cc. of water.

Remarks.—Creosote water is not a permanent preparation, for the filtering solution, on standing, becomes turbid, and deposits some of the decomposition products of the creosote in the form of a smeary tar.

For this reason the pharmacopœia directs it to be freshly prepared.

Dose.—8 Cc. (2 fluidrachms).

AQUA DESTILLATA. Distilled Water.

This official, being a chemical rather than a pharmaceutical, will be discussed in Part III.

AQUA FŒNICULI. Fennel Water.

Recipe.—Made by triturating 2 Cc. Oil of Fennel with purified talc and water to make 1000 Cc. Details identical with recipe for anise water.

Dose.—16 Cc. (4 fluidrachms).

AQUA HAMAMELIDIS. Hamamelis Water.

<i>Recipe.</i> —Hamamelis Bark, ten thousand grammes.....	10000 Gm.
Water, twenty thousand cubic centimeters.....	20000 Cc.
Alcohol, fifteen hundred cubic centimeters.....	1500 Cc.
To make ten thousand cubic centimeters..	10000 Cc.

Macerate the Hamamelis Bark in the Water during twenty-four hours; then distil until *eighty-five hundred cubic centimeters* of distillate are obtained, add the Alcohol, and mix thoroughly.

If 1 Cc. of Hamamelis Water be added to 5 Cc. of sulphuric acid containing a little salicylic acid in solution, no red color should appear (absence of *formaldehyde*).

Remarks.—This new official is what is commonly called *distilled extract of witch hazel*, and has attained widespread popularity through the persistent advertising of a certain proprietary form of it. Its therapeutic value is doubtful, many authorities claiming that its sole virtue resides in the alcohol it contains. The recipe given above is impossible to any save those residing where the plant abounds, but it is of value in an effort to establish a definite basis of manufacture, each witch hazel distiller having been, until now, a law unto himself. Of particular importance to the retailer is the pharmacopœial demand that this product contain 15 per cent. *grain* alcohol (ethyl alcohol of the pharmacopœia), and that no formaldehyde be present. Many distillers have of late used wood alcohol, and in the crusade against the use of this poison in drugs several honest pharmacists have been prosecuted for dispensing methylated witch hazel—another illustration of the importance of the testing, by the pharmacist, of every product sold by him under his label and not made in his laboratory.

Dose.—8 Cc. (2 fluidrachms).

AQUA HYDROGENII DIOXIDI. Solution of Hydrogen Dioxide.

This is called in the pharmacopœia the *solution* of the dioxide, it being the only case in which the English pharmacopœial name of the preparation places it in a class different from the Latin name. The reason that it is called the *solution* of hydrogen dioxide by the pharmacopœia is because it has been sold in commerce always under the name of solution. Whether it should be called "*Aqua Hydrogenii Dioxidii*" is questionable, since hydrogen dioxide is not markedly volatile.

Hydrogen dioxide solution contains about 3 per cent. of pure H_2O_2 . The process of its manufacture is given on p. 387.

AQUA MENTHÆ PIPERITÆ. Peppermint Water.

Recipe.—1000 Cc. is made from 2 Cc. Oil of Peppermint by process exactly the same as that for making anise water—trituration with purified talc.

Dose.—16 Cc. (4 fluidrachms).

AQUA MENTHÆ VIRIDIS. Spearmint Water.

Recipe.—1000 Cc. is made from 2 Cc. Oil of Spearmint, exactly as in the recipe for anise water.

Dose.—16 Cc. (4 fluidrachms).

AQUA ROSÆ. Rose Water.

Recipe.—By dilution of stronger Rose Water with equal quantity of water, as in recipe for orange flower water.

Dose.—16 Cc. (4 fluidrachms).

AQUA ROSÆ FORTIOR. Stronger Rose Water.

Water saturated with volatile oil of Rose petals, obtained by distillation.

Stronger Rose Water should be kept in bottles loosely stoppered with a pledget of purified cotton, and in a dark place.

Stronger Rose Water should be colorless and clear, not mucilaginous, and should have the odor of roses, free from empyreuma. It should give no reaction with hydrogen sulphide T.S. or ammonium sulphide T.S. (absence of *metallic impurities*).

Manufacture.—No recipe given by the pharmacopœia, but it is a triple distilled water.

Remarks.—The two varieties of rose water are analogous to the two official orange flower waters already discussed.

Dose.—8 Cc. (2 fluidrachms).

Medical Properties of Waters.—The medical properties of most pharmaceutic preparations are those of the drugs from which they are obtained, and therapeutic consideration will be found in the proper place under the drug which is the leading ingredient.

In some few cases, however, the preparation consists of a blending of ingredients giving different action than the constituent drugs; or possibly may be a commercial, ready-made article, the constituents of which are not official. In such cases, illustrated in this chapter in orange flower water, the medical properties will be discussed at the time the preparation is considered.

Waters as a class are usually such diluted preparations that they are scarcely used as medicines, but are generally employed as vehicles. As notable exceptions, however, must be cited such waters as the ammonia waters, chlorine water, hydrogen dioxide water; these, being rather powerful preparations and having special uses, will be considered in their place among the chemicals.

DOSES OF OFFICIAL WATERS

1 Cc. (15 minims).	Ammonia water.
4 Cc. (1 fluidrachm).	Bitter almond water, solution of hydrogen dioxide.
8 Cc. (2 fluidrachms).	Camphor water, creosote water, hamamelis water, stronger orange flower water, stronger rose water.
16 Cc. (4 fluidrachms).	Anise water, orange flower water, chloroform water, cinnamon water, fennel water, peppermint water, spearmint water, rose water.

Stronger ammonia water should never be dispensed for administration by a patient, it being recognized by the pharmacopœia merely as a standard form of ammonia which can be diluted to a 10 per cent. strength when required.

SPIRITS

A spirit is *an alcoholic solution of a volatile substance*, and, therefore, they differ from waters only in the solvent employed. For this reason a number of spirits are made from substances which are also used in the manufacture of waters. Practically, all the volatile oils repre-

sented in the list of aromatic waters are found in alcoholic solutions among the spirits. The number of spirits, however, is larger than that of the waters, due to the superior solubility of oils in alcohol. Moreover, the amount of oils found in spirits is much greater than that found in waters, most of the spirits containing from 5 to 10 per cent. or even 20 per cent. of the oil.

TABLE OF OFFICIAL SPIRITS

<i>Manufacture.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparation.</i>
	Spirit of ammonia, aromatic. Spirit of anise. Spirit of bitter almond. Spirit of camphor. Spirit of chloroform. Spirit of cinnamon. Spirit of ether. Spirit of ether, compound. Spirit of gaultheria. Spirit of glyceryl trinitrate. Spirit of juniper. Spirit of juniper, compound. Spirit of lavender. Spirit of myrcia (U. S. P. 1890). Spirit of orange, compound. Spirit of phosphorus (U. S. P. 1890).	
<i>Simple solution or dilution.</i>		Spirit of nitrous ether.
<i>Gaseous solution</i>	Spirit of ammonia.	
<i>Solution with maceration</i> ...	Spirit of lemon (U. S. P. 1890). Spirit of peppermint. Spirit of spearmint.	
<i>Distillation</i>	Whisky. Brandy.	

As is seen in the above chart, the official spirits are made in four different ways:

Simple Solution or Dilution.—This process is simply adding and dissolving the medicated substance in alcohol. (In this group we find fifteen of the twenty official spirits.)

Gaseous Solution.—One official spirit—spirit of ammonia—is made by the passing of the gas into alcohol, and the details will be left for explanation in the pharmacopœial recipe for this spirit.

Solution by Maceration.—Two official spirits are made in this way—peppermint and spearmint. In each case the flavoring constituent is a volatile oil, but in deference to the wishes of the public who desire a spirit colored like the drug from which it is made, the pharmacopœia directs that, after the oil is added to the alcohol, it is to be macerated with some of the drug until the color of the latter has been absorbed by the alcohol.

Distillation.—Two official spirits are made by distillation, and the technical details of each operation will be found on page 653.

Two official spirits may be considered as made by *chemical action*—spirit of glyceryl trinitrate and spirit of nitrous ether. In one case, however, no direction is given by the pharmacopœia for the preparation of the chemical (glyceryl trinitrate), it being a task considered

too dangerous for the ordinary drug-store; while in the case of the spirit of nitrous ether, the spirit is really made by dissolving the finished ethyl nitrite in alcohol, and the process may, therefore, be considered as a case of simple solution or dilution. For this reason both of these preparations are found in the chart given above under the head of simple solution or dilution.

SPECIAL NOTES ON OFFICIAL SPIRITS

SPIRITUS ÆTHERIS. Spirit of Ether.

<i>Recipe.</i> —Ether, three hundred and twenty-five cubic centimeters.	325 Cc.
Alcohol, six hundred and seventy-five cubic centimeters.	675 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them.

Remarks.—This is made by the simple solution of ether and alcohol, and is sold in Germany under the name of Hoffman's drops.

Dose.—4 Cc. (1 fluidrachm).

SPIRITUS ÆTHERIS COMPOSITUS. Compound Spirit of Ether.

<i>Recipe.</i> —Ether, three hundred and twenty-five cubic centimeters.	325 Cc.
Alcohol, six hundred and fifty cubic centimeters.....	650 Cc.
Ethereal Oil, twenty-five cubic centimeters.....	25 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them.

Remarks.—Compound spirit of ether is usually called "Hoffman's anodyne." It contains, besides ether and alcohol, ethereal oil—the indefinite preparation referred to on p. 664. As commercial ethereal oil is notoriously adulterated, it should be made by the retail pharmacist.

Dose.—4 Cc. (1 fluidrachm).

SPIRITUS ÆTHERIS NITROSI. Spirit of Nitrous Ether.

A full explanation of this important preparation, commonly called *sweet spirits of nitre*, will be found on p. 661.

SPIRITUS AMMONIÆ. Spirit of Ammonia.

This spirit is made by gaseous solution and contains 10 per cent. of ammonia gas, like ammonia water. The recipe and other details of manufacture will be considered on p. 485, under the preparations of ammonia.

SPIRITUS AMMONIÆ AROMATICUS. Aromatic Spirit of Ammonia.

This important pharmaceutical can best be considered under the Ammonia Compounds (p. 490).

SPIRITUS AMYGDALÆ AMARÆ. Spirit of Bitter Almond.

<i>Recipe.</i> —Oil of Bitter Almond, ten cubic centimeters.....	10 Cc.
Alcohol, eight hundred cubic centimeters.....	800 Cc.
Distilled Water, a sufficient quantity,	

To make one thousand cubic centimeters.. 1000 Cc.

Dissolve the Oil in the Alcohol, and add enough Distilled Water to make the product measure one thousand cubic centimeters.

Remarks.—The synonym for this preparation is *essence of bitter almond*, and it might be here stated that the so-called “essences,” or flavoring extracts, represent alcoholic preparations of aromatic principles. Thus, among the spirits we find the essence of almond, essence of peppermint, essence of spearmint, and essence of lemon of the pharmacopœia of 1890. Essence of ginger and essence of vanilla are respectively tincture of ginger and tincture of vanilla. The spirit of bitter almond, it might be worth noting, contains about 20 per cent. of water.

Dose.—0.5 Cc. (8 minims).

SPIRITUS ANISI. Spirit of Anise.

Recipe.—100 Cc. Oil of Anise dissolved in 900 Cc. Alcohol. Details like recipe for spirit of ether.

Remarks.—This is a 10 per cent. preparation made by simple solution. Bear in mind that the alcohol of the present pharmacopœia is equivalent to the deodorized alcohol (Cologne Spirits) of the pharmacopœia of 1890, and is devoid of the unpleasant odor which ordinary alcohol leaves on evaporation. In the preparation of essences made from fine oils this grade of alcohol must be used instead of ordinary alcohol.

Dose.—4 Cc. (1 fluidrachm).

SPIRITUS AURANTII COMPOSITUS. Compound Spirit of Orange.

Recipe.—Oil of Orange Peel, two hundred cubic centimeters. 200 Cc.
 Oil of Lemon, fifty cubic centimeters. 50 Cc.
 Oil of Coriander, twenty cubic centimeters. 20 Cc.
 Oil of Anise, five cubic centimeters. 5 Cc.
 Alcohol, a sufficient quantity,

To make one thousand cubic centimeters. . . 1000 Cc.

Mix them. Keep the product in completely filled, well-stoppered bottles, in a cool and dark place.

Remarks.—This preparation, used as the flavor in *aromatic elixir* (p. 205), must be made of the best oils and alcohol if it is to be satisfactory. The provision as to storing given above is to prevent the oils from growing terebinthinate.

SPIRITUS CAMPHORÆ. Spirit of Camphor.

Recipe.—Camphor, one hundred grammes. 100 Gm.
 Alcohol, a sufficient quantity,

To make one thousand cubic centimeters. . . 1000 Cc.

Dissolve the Camphor in eight hundred cubic centimeters of Alcohol, filter through paper, and pass enough Alcohol through the filter to make the product measure one thousand cubic centimeters.

Remarks.—Note that the recipe of the pharmacopœia does not direct the addition of water, and that such addition would be considered by pure food officials as an adulteration.

Dose.—1 Cc. (15 minims).

SPIRITUS CHLOROFORMI. Spirit of Chloroform.

Recipe.—Made by dissolving 60 Cc. Chloroform in 940 Cc. Alcohol. Details similar to the recipe for spirit of ether.

Remarks.—Note that this is a 6 per cent. preparation.

Dose.—2 Cc. (30 minims).

SPIRITUS CINNAMOMI. Spirit of Cinnamon.

Recipe.—Made by dissolving 100 Cc. Oil of Cinnamon in 900 Cc. Alcohol, with spirit of ether.

Dose.—2 Cc. (20 minims).

SPIRITUS FRUMENTI. Whisky.

A discussion of this distillate from fermented grain will be given on p. 653.

SPIRITUS GAULTHERIÆ. Spirit of Gaultheria.

Recipe.—Oil of Gaultheria, 50 Cc.; Alcohol, 950 Cc., dissolved as in making spirit of ether.

Remarks.—This is commonly called *essence of wintergreen*.

Dose.—2 Cc. (30 minims).

SPIRITUS GLYCERYLIS NITRATIS. Spirit of Glyceryl Trinitrate.

This very important official, Spirit of Nitroglycerin, is fully described on p. 681. Here mention must be made, however, of the pharmacopœial warning, that even tasting a small quantity of it is liable to produce violent headache; also mention must be made that, since it contains the very explosive nitroglycerin, great care must be taken to prevent the spilling of the spirit. When the alcohol has evaporated, nitroglycerin remains, and may be brought to an explosion by simple concussion. If the spirit is spilled on the floor or elsewhere, it should be immediately treated with a diluted solution of soda, which will break it up into the two harmless substances—glycerin and sodium nitrate—and these can then be washed up.

SPIRITUS JUNIPERI. Spirit of Juniper.

Recipe.—Oil of Juniper, 50 Cc.; Alcohol, 950 Cc., dissolved as in making spirit of ether.

Dose.—2 Cc. (30 minims).

SPIRITUS JUNIPERI COMPOSITUS. Compound Spirit of Juniper.

<i>Recipe.</i> —Oil of Juniper, eight cubic centimeters.....	8 Cc.
Oil of Caraway, one cubic centimeter.....	1 Cc.
Oil of Fennel, one cubic centimeter.....	1 Cc.
Alcohol, fourteen hundred cubic centimeters.....	1400 Cc.
Water, a sufficient quantity,	

To make two thousand cubic centimeters.. 2000 Cc.

Dissolve the Oils in the Alcohol, and gradually add enough Water to make the product measure two thousand cubic centimeters.

Average Dose.—8 Cc. (2 fluidrachms).

Remarks.—The preparation is intended as a substitute for gin, which is made by distilling fermented rye and malt, macerating the distillate with juniper berries, and redistilling.

Dose.—8 Cc. (2 fluidrachms).

SPIRITUS LAVANDULÆ. Spirit of Lavender.

Recipe.—Oil of Lavender Flowers, 50 Cc.; Alcohol, 950 Cc., dissolved as in making spirit of ether.

Dose.—2 Cc. (30 minims).

SPIRITUS LIMONIS (U. S. P. 1890). Spirit of Lemon.

This spirit, replaced in the present pharmacopœia by *tincture of lemon peel* (p. 230), is prepared by dissolving 50 Cc. oil of lemon in enough deodorized alcohol to make 1000 Cc., and macerating the mixture with 50 Gm. grated lemon peel in order to give a color. The spirit, therefore, contains 5 per cent. of oil, and it is hardly necessary to say that this oil should be entirely free from terebinthinate odor. The finished product is called *essence of lemon*.

SPIRITUS MENTHÆ PIPERITÆ. Spirit of Peppermint.

Recipe.—Oil of Peppermint, one hundred cubic centimeters. . . . 100 Cc.
Peppermint, bruised, ten grammes 10 Gm.
Alcohol, a sufficient quantity,
To make one thousand cubic centimeters. . . 1000 Cc.

Dissolve the Oil of Peppermint in nine hundred cubic centimeters of Alcohol, add the Peppermint, and allow it to macerate for twenty-four hours. Then filter through paper, and add, through the filter, enough Alcohol to make the Spirit measure one thousand cubic centimeters.

Remarks.—This is a 10 per cent. preparation of the oil of peppermint made with alcohol, and macerated with sufficient peppermint leaves to give it a green color. This is the popular *essence of peppermint*.

Dose.—2 Cc. (30 minims).

SPIRITUS MENTHÆ VIRIDIS. Spirit of Spearmint.

Recipe.—Oil of Spearmint, 100 Cc.; Spearmint (bruised), 10 Gm.; Alcohol, to make 1000 Cc. Make exactly as in recipe for spirit of peppermint.

Dose.—2 Cc. (30 minims).

SPIRITUS MYRCIÆ (U. S. P. 1890). Spirit of Myrcia.

This preparation, dropped in the last pharmacopœial revision, is an excellent form of *bay-rum*. It was made by dissolving 16 Cc. oil of myrcia, 1 Cc. oil of orange peel, 1 Cc. oil of pimenta, in 1220 Cc. deodorized alcohol, and then adding water to make 2000 Cc., and filtering.

SPIRITUS PHOSPHORI (U. S. P. 1890). Spirit of Phosphorus.

This product, dropped in the last pharmacopœial revision, was interesting because made with absolute alcohol and by the process of upright condensation (see p. 102). This process was employed to save undue evaporation of alcohol, it being necessary to digest the phosphorus in boiling alcohol for several hours in order to dissolve it. When this process is carried on in an ordinary flask, a large amount of alcohol would be lost by evaporation, but if the upright condenser is inserted in the neck of the flask, the vapor of alcohol passing through the cold condensing tube is converted into liquid, and drops back into the flask from which it has just emerged. Spirit of phosphorus was official because an ingredient of the *elixir of phosphorus* of the pharmacopœia of 1890.

SPIRITUS VINI GALLICI. Brandy.

Manufacture, alcoholic strength, and use of this substance will be fully given on p. 653. Here it need only be said that it is one of the two official spirits prepared by distillation.

DOSES OF SPIRITS

0.05 Cc. (1 minim).....	Spirit of glyceryl trinitrate.
0.5 Cc. (8 minims).....	Spirit of bitter almond.
1.0 Cc. (15 minims).....	Spirits of ammonia and of camphor.
2.0 Cc. (30 minims).....	Spirits of nitrous ether, ammonia aromatic, chloroform, cinnamon, gaultheria, juniper, lavender, peppermint, and spearmint.
4 Cc. (1 fluidrachm).....	Spirits of ether, ether compound, and anise.
8 Cc. (2 fluidrachms).....	Spirit of juniper compound.

CHAPTER XII**SOLUTIONS, MUCILAGES, SYRUPS, AND HONEYS****SOLUTIONS**

SOLUTIONS constitute that class of pharmaceuticals defined as "*aqueous solutions of non-volatile bodies.*" This definition is redundant, inasmuch as we say that a "solution" (the class of pharmaceuticals) is a "solution" (that is, a product of the physical phenomenon called solution, which has been discussed on p. 117). In other words, the student at the outset should get a clear idea of the three meanings of the word "solution." First, the physical phenomenon of molecular subdivision resulting in a homogeneous fluid; second, the physical group of solutions, those homogeneous fluids produced by molecular subdivision, and including such pharmaceuticals as waters, spirits, glycerites, tinctures, etc.; and, third, the narrow pharmaceutic

“solution,” which includes only that class of physical solutions which have water as the solvent and a non-volatile body as dissolved substance.

A pharmacopœial solution is an aqueous preparation, free from sugar (exception, Solution of Magnesium Citrate), made by the ordinary process of solution. Some writers divide this class into simple and chemical solutions, while others oppose such a distinction, claiming there is no real difference in the liquefaction occurring in, say, solution of arsenous acid and in solution of ferric acetate. The latter class of writers claim that the chemical combination occurring in the formation of a so-called “chemical solution” has nothing to do with the act of solution, the chemical reaction first occurring, then being followed by the solution of the newly formed substance or of the ions of which it is composed. (See p. 119.) There is much truth in the argument that the arsenous acid in the first solution cited above is produced by chemical reaction, as much as is the ferric acetate in the other preparation; that the fact whether the substance is dissolved at the moment of formation or after some time has elapsed makes little difference.

In this book we will divide solutions into galenic preparations (simple solutions) and into chemical preparations (chemical solutions), for the same reason that we group all pharmaceuticals into the two classes; that in Part II. we will discuss only these preparations made without chemical action, leaving those in which a chemical action occurs for consideration in Part III.

The manufacture of solutions is simple or complex, according as the preparation is a galenic or a chemical. Galenic solutions are prepared by simply dissolving the substance in an appropriate quantity of water in either a flask or a bottle, and dissolving by agitation, or by first reducing the substance to a fine powder by trituration in a mortar, and gradually adding water to the substance in the mortar.

The preparation of the so-called “simple solutions” is an easy matter—so easy that the average druggist is grossly careless with them. They should be elegant preparations; hence the careful pharmacist is generous with his filter-paper, having all his solutions (save possibly lead water) as clear as crystal. This applies to all solutions, and especially to extemporaneous prescriptions. Have all preparations, intended to be clear, fairly sparkling, for the only method possessed by the public of judging the skill of the pharmacist is the appearance of his preparations and the neatness of his packages.

The chemical solution, on the other hand, usually requires a high degree of skill in its manufacture, and the description of this class had best be left until we consider the substance from the chemical aspect in Part III. The following table gives a list of officials arranged as galenic or chemical:

TABLE OF OFFICIAL SOLUTIONS

<i>Manufacture.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparations.</i>
All galenics are made by simple solution.	Solution of arsenous acid.	Solution of ammonium acetate.
	Antiseptic solution.	Solution of calcium hydroxide.
	Solution of arsenous and mercuric iodide.	Solution of chlorine, compound.
	Solution of formaldehyde.	Solution of cresol, compound
	Solution of iodine, compound.	Solution of ferric chloride.
	Solution of lead subacetate, diluted.	Solution of ferric citrate (U. S. P. 1890).
	Solution of potassium hydroxide.	Solution of ferric nitrate (U. S. P. 1890).
	Solution of sodium arsenate.	Solution of ferric subsulphate.
	Solution of sodium hydroxide.	Solution of ferric sulphate.
	Solution of sodium phosphate, compound.	Solution of iron and ammonium acetate.
		Solution of lead subacetate.
		Solution of mercuric nitrate.
		Solution of magnesium citrate.
		Solution of potassa (U. S. P. 1890).
		Solution of potassium arsenite.
		Solution of potassium citrate.
		Solution of soda (U. S. P. 1890).
		Solution of chlorinated soda.
		Solution of sodium silicate (U. S. P. 1890).
		Solution of zinc chloride.

SPECIAL NOTES ON OFFICIAL SOLUTIONS

LIQUOR ACIDI ARSENOSSI. Solution of Arsenous Acid.

An aqueous solution, which should contain Arsenous Acid, corresponding in amount to 1 per cent. of arsenic trioxide [$\text{As}_2\text{O}_3 = 196.44$].

Recipe.—Arsenic Trioxide, ten grammes 10 Gm.
 Diluted Hydrochloric Acid, fifty grammes..... 50 Gm.
 Distilled Water, a sufficient quantity,
 To make one thousand grammes.... 1000 Gm.

Mix the Diluted Hydrochloric Acid with *two hundred and fifty grammes* of Distilled Water, in a tared porcelain dish, add the Arsenic Trioxide, and boil the mixture until the Arsenic Trioxide is dissolved. Then add enough Distilled Water to make the product weigh *one thousand grammes*. Filter through paper.

A clear, colorless liquid, odorless, having an acidulous taste and an acid reaction. If to 24.6 Gm. of Solution of Arsenous Acid about 2 Gm. of sodium bicarbonate and 100 Cc. of water be added, not less than 50 Cc. of tenth-normal iodine V.S. should be required to produce a permanent yellow tint (corresponding to 1 Gm. of arsenic trioxide in 100 Gm. of the Solution).

Remarks.—This is a 1 per cent. solution of arsenous acid in water, containing 5 per cent. of diluted hydrochloric acid. The preparation was formerly called *solution of arsenic chloride*, it being supposed that the hydrochloric acid combined with the arsenous acid with the formation of the chloride. This view is not generally held, however, at the present time, the function of the hydrochloric acid being supposed as merely rendering the arsenous acid more soluble.

Dose.—0.2 Cc. (3 minims).

LIQUOR AMMONII ACETATIS. Solution of Ammonium Acetate.

This useful preparation, known as *Spirit of Mindererus*, will be discussed on p. 487.

LIQUOR ANTISEPTICUS. Antiseptic Solution.

<i>Recipe.</i> —Boric Acid, twenty grammes.....	20.00 Gm.
Benzoic Acid, one gramme.....	1.00 Gm.
Thymol, one gramme.....	1.00 Gm.
Eucalyptol, one-fourth cubic centimeter.....	0.25 Cc.
Oil of Peppermint, one-half cubic centimeter.....	0.50 Cc.
Oil of Gaultheria, one-fourth cubic centimeter....	0.25 Cc.
Oil of Thyme, one-tenth cubic centimeter.....	0.10 Cc.
Alcohol, two hundred and fifty cubic centimeters..	250.00 Cc.
Purified Talc, twenty grammes.....	20.00 Gm.
Water, a sufficient quantity,	

To make one thousand cubic centimeters... 1000.00 Cc.

Dissolve the Boric Acid in seven hundred cubic centimeters of Water, and the Benzoic Acid in one hundred and fifty cubic centimeters of Alcohol, and pour the aqueous solution into the alcoholic solution. Then dissolve, in a mortar, the Thymol in the Eucalyptol and Oils of Peppermint, Gaultheria, and Thyme; thoroughly incorporate the Purified Talc, and add, with constant trituration, the solution first prepared. Allow the mixture to stand, with occasional agitation, during forty-eight hours, filter, add one hundred cubic centimeters of Alcohol to the clear filtrate, and a sufficient quantity of Water to make the finished product measure one thousand cubic centimeters.

Remarks.—This new official is an elegant preparation similar to the many antiseptic solutions exploited under trade names. To secure an entirely satisfactory product it is essential that the oils employed be of the finest quality.

Medical Properties.—Used for the many purposes for which a harmless antiseptic can be employed—a gargle for sore throat, a mouth-wash, and internally for gastric disturbances. For the latter purpose, however, it should be used with caution.

Dose.—4 Cc. (1 fluidrachm).

LIQUOR ARSENI ET HYDRARGYRI IODIDI. Solution of Arsenous and Mercuric Iodides.

An aqueous solution, which should contain not less than 1 per cent. of Arsenous Iodide and 1 per cent. of Mercuric Iodide.

<i>Recipe.</i> —Arsenous Iodide, ten grammes.....	10 Gm.
Red Mercuric Iodide, ten grammes.....	10 Gm.
Distilled Water, a sufficient quantity,	

To make one thousand grammes.. 1000 Gm.

Rub the Arsenous Iodide and Red Mercuric Iodide together in a mortar, add one hundred and fifty cubic centimeters of Distilled Water, and continue the trituration until solution is effected. Filter the solution, and pass sufficient Distilled Water through the filter to make the product weigh one thousand grammes. Mix thoroughly.

A clear, colorless or pale yellowish liquid, without odor, and having a disagreeable metallic taste.

Remarks.—This preparation has as its synonym *Donovan's Solution*, and contains 1 per cent. of arsenous iodide and 1 per cent. of mercuric iodide dissolved in water. The pharmacopœia directs that Donovan's solution should be not more than a pale yellow color;

but on standing this solution becomes red, and such red solutions should not be dispensed, as it is supposed to contain traces of free iodine. This may be prevented by placing a globule of mercury in the container holding the solution.

LIQUOR CALCIS. Solution of Calcium Hydroxide.

This well-known preparation, commonly called *Lime Water*, will be considered among the other preparations of calcium (p. 506). In the present system of grouping pharmaceuticals the old Latin name *Aqua Calcis* is a misnomer, since its active constituent, calcium hydroxide, is a non-volatile solid, while official waters are solutions of volatile bodies.

LIQUOR CHLORI COMPOSITUS. Compound Solution of Chlorine.

As already mentioned, the new official replaces the formerly official chlorine water, and it will be discussed among the preparations of chlorine (p. 371).

LIQUOR CRESOLIS COMPOSITUS. Compound Solution of Cresol.

This new official, similar to but not identical with the patent preparations, *creolin* and *lysol*, will be discussed among the cresol derivatives (p. 773).

LIQUOR FERRI CHLORIDI. Solution of Ferric Chloride.

LIQUOR FERRI ET AMMONII ACETATIS. Solution of Iron and Ammonium Acetate.

LIQUOR FERRI SUBSULPHATIS. Solution of Ferric Subsulphate.

LIQUOR FERRI SULPHATIS. Solution of Ferric Sulphate.

These four chemical solutions of iron will be discussed in the chapter devoted to that element (Chapter XXXI).

LIQUOR FORMALDEHYDI. Solution of Formaldehyde.

This 37 per cent. solution of the gas formaldehyde, sometimes called *Formalin*, will be discussed under Formaldehyde (p. 647).

LIQUOR HYDRARGYRI NITRATIS. Solution of Mercuric Nitrate.

This heaviest of all official solutions will be considered on p. 619.

LIQUOR IODI COMPOSITUS. Compound Solution of Iodine.

An aqueous solution, which should contain not less than 5 per cent. of iodine and 10 per cent. of potassium iodide.

Recipe.—Iodine, five grammes..... 5 Gm.
Potassium Iodide, ten grammes..... 10 Gm.
Distilled Water, a sufficient quantity,
To make one hundred grammes.. 100 Gm.

Dissolve the Iodine and Potassium Iodide in a sufficient quantity of Distilled Water to make the product weigh one hundred grammes. Keep the Solution in glass-stoppered bottles.

For complete decolorization 6.3 Gm. of the Solution should require not less than 24.75 Cc. of tenth-normal sodium thiosulphate V.S. (each Cc. of the tenth-normal sodium thiosulphate V.S. corresponding to 0.2 per cent. of Iodine).

Remarks.—The synonym of this compound is *Lugol's solution*, which should not be confounded with *Dobell's solution* (p. 459), as it was in one case when a pharmacist translated the poor writing of a physician calling for Dobell's solution into Lugol's solution, which was accordingly sprayed into the throat, with exceedingly irritating results.

Compound solution of iodine contains 5 per cent. of iodine dissolved in water by means of 10 per cent. of potassium iodide. This proves the interesting fact that while iodine scarcely dissolves in water, it is freely soluble in solution of potassium iodide.

Dose.—0.2 Cc. (3 minims).

LIQUOR MAGNESII CITRATIS. Solution of Magnesium Citrate.

This popular and valuable official, sometimes called "Purgative Lemonade," will be discussed on p. 500.

LIQUOR PLUMBI SUBACETATIS. Solution of Lead Subacetate.

Discussed on p. 601.

LIQUOR PLUMBI SUBACETATIS DILUTUS. Diluted Solution of Lead Subacetate.

An aqueous liquid, which should contain about 1 per cent. of Lead Subacetate.

Recipe.—Solution of Lead Subacetate, forty grammes. 40 Gm.

Distilled Water, a sufficient quantity,

To make one thousand grammes. . . 1000 Gm.

Mix the Solution of Lead Subacetate with enough Distilled Water, previously boiled and cooled, to make the product weigh one thousand grammes. Keep the Solution in well-stoppered bottles.

Remarks.—This official is called *lead water*, and contains but 4 parts of solution of lead subacetate to 100 parts finished solution; hence it is much weaker. The pharmacopœia provides that the solution of lead subacetate be diluted with distilled water which has been previously boiled and cooled. The boiling in this case is to rid the water of its dissolved carbonic acid, for if the unboiled water is brought into contact with the solution of lead subacetate, a precipitation of lead carbonate occurs, with corresponding loss in the lead strength of the official preparation. In order to prevent the formation of this lead carbonate in the official product the pharmacopœia directs that the solution be kept in well-stoppered bottles. Lead water is best dispensed opalescent, to avoid the danger of confusing this poisonous preparation with the harmless "lime water."

Medical Properties.—Used exclusively externally as a soothing wash in frost-bite, ivy-poisoning, etc. Hence no dose is given by the pharmacopœia.

LIQUOR POTASSII ARSENITIS. Solution of Potassium Arsenite.

LIQUOR POTASSII CITRATIS. Solution of Potassium Citrate.

LIQUOR POTASSII HYDROXIDI. Solution of Potassium Hydroxide.

Can best be studied among the derivatives of potassium (Chapter XXV.).

LIQUOR SODÆ CHLORINATÆ. Solution of Chlorinated Soda.

See p. 393.

LIQUOR SODII ARSENATIS. Solution of Sodium Arsenate.

An aqueous solution, which should contain Sodium Arsenate corresponding in amount to not less than 1 per cent. of Exsiccated Sodium Arsenate.

Recipe.—**Exsiccated Sodium Arsenate**, one gramme..... 1 Gm.
Distilled Water, a sufficient quantity,
 To make one hundred grammes.. 100 Gm.

Dissolve the Exsiccated Sodium Arsenate in a sufficient quantity of Distilled Water to make the product weigh *one hundred grammes*.

The Solution should conform to the reactions and tests for an aqueous solution of the salt given under *Sodii Arsenas Exsiccatus*.

Remarks.—This is a 1 per cent. solution of sodium arsenate in water. The sodium arsenate must, however, be previously deprived of its water of crystallization; that is, it must be the exsiccated sodium arsenate of the present pharmacopœia. This preparation is one of the four arsenical solutions directed by the pharmacopœia, the others being solution of arsenous acid, solution of arsenous and mercuric iodides, and solution of potassium arsenite, the latter being considered among the chemical solutions.

It is well to bear in mind that the strength of these four arsenical solutions, noting that while all of them are 1 per cent., it does not mean that each contains 1 per cent. of arsenic trioxide, U. S. P. Two of these, solution of arsenous acid and solution of potassium arsenite, contain 1 per cent. of arsenic trioxide; solution of arsenic and mercuric iodide contains 1 per cent. of arsenic iodide, while in solution of sodium arsenate the arsenical compound is sodium arsenate.

Dose.—0.2 Cc. (3 minims).

LIQUOR SODII HYDROXIDI. Solution of Sodium Hydroxide.

While this preparation, commonly called *solution of soda*, is directed by the present pharmacopœia to be made by simple solution of sodium hydroxide in water, former pharmacopœias directed its manufacture by chemical action. Since the chemistry of this solution is most interesting, discussion will be deferred until we can take up the chemical side (p. 456).

The same statement applies with equal force to solution of potassium hydroxide.

LIQUOR SODII PHOSPHATIS COMPOSITUS. Compound Solution of Sodium Phosphate.

This galenic can best be considered under Sodium Phosphate (p. 471).

LIQUOR ZINCI CHLORIDI. Solution of Zinc Chloride.

See p. 523.

THE DOSES OF OFFICIAL SOLUTIONS

0.1 Cc. ($1\frac{1}{2}$ minims).....	Solutions of arsenous and mercuric iodide and of ferric chloride.
0.2 Cc. (3 minims).....	Solutions of arsenous acid, ferric subsulphate, iodine compound, potassium arsenite, and sodium arsenate.
1 Cc. (15 minims).....	Solutions of potassium hydroxide, chlorinated soda, and sodium hydroxide.
4 Cc. (1 fluidrachm).....	Antiseptic solution and solution of chlorine compound.
8 Cc. (2 fluidrachms).....	Solution of sodium phosphate compound.
16 Cc. (4 fluidrachms).....	Solutions of ammonium acetate, calcium hydroxide, iron and ammonium acetate, and potassium citrate.
360 Cc. (12 fluidounces).....	Solution of magnesium citrate.

No doses are assigned the official solutions not mentioned in the above list.

MUCILAGES

The mucilages are *aqueous preparations containing viscid substances in solution or suspension*, and from a strictly physical point of view mucilages should scarcely be grouped among the solutions.

All mucilages should be freshly prepared, as their mucilaginous constituents when diluted with water are usually prone to fermentation.

One of the official mucilages is a jelly and the others are liquids. Two official mucilages are prepared with hot water—mucilage of tragacanth and the mucilage of elm. The other two are prepared with cold water—mucilage of acacia and mucilage of sassafras pith. All four are galenic preparations.

SPECIAL NOTES ON OFFICIAL MUCILAGES**MUCILAGO ACACIÆ. Mucilage of Acacia.**

<i>Recipe.</i> —Acacia, in small fragments, three hundred and forty grammes.....	340 Gm.
Lime Water, three hundred and thirty grammes.....	330 Gm.
Water, a sufficient quantity,	
To make one thousand grammes. .	1000 Gm.

Wash the Acacia with cold Water, and allow it to drain. Add the Lime Water to it, and enough Water to make the mixture weigh *one thousand grammes*, agitate or stir it occasionally until the Acacia is dissolved, and strain. Keep the product in well-stoppered, completely filled bottles, in a cool place.

Remarks.—This preparation contains from 3 to 4 per cent. of washed gum arabic dissolved in lime water and water, and the pharmacopœia directs that the gum arabic be in the form of small pieces and not in a powder. For general prescription purposes, however, especially when the mucilage of gum arabic is directed as one of the several ingredients, the powdered gum arabic is generally used, its solution being much quicker than the gum arabic in lumps. The lime water is added to facilitate solution of the gum, the free arabic acid it sometimes contains being thus converted into the more soluble calcium arabate. The lime water also acts, to a certain extent, as a preservative.

In preparing large quantities of the mucilage of acacia, as in making mucilage to sell as an adhesive, the solution of the gum arabic can be facilitated by placing the gum in a muslin bag and suspending it on the surface of the water contained in the crock. In this way the gum arabic can be easily dissolved in the course of a few hours by means of a circulatory solution.

Dose.—16 Cc. (4 fluidrachms).

MUCILAGO SASSAFRAS MEDULLÆ. Mucilage of Sassafras Pith.

Recipe.—Sassafras Pith, two grammes..... 2 Gm.
Water, one hundred cubic centimeters..... 100 Cc.

Macerate the Sassafras Pith in the Water during three hours, and strain without expression. This preparation should be freshly made when wanted.

Remarks.—This is prepared by macerating 2 per cent. of sassafras pith during three hours and then straining it.

The mucilage of sassafras pith is used as a basis of some eye remedies and is rarely given internally.

Dose.—16 Cc. (4 fluidrachms).

MUCILAGO TRAGACANTHÆ. Mucilage of Tragacanth.

Recipe.—Tragacanth, six grammes..... 6 Gm.
Glycerin, eighteen grammes..... 18 Gm.
Water, a sufficient quantity,
To make one hundred grammes.. 100 Gm.

Mix the Glycerin with *seventy-five cubic centimeters* of Water in a tared vessel, heat the mixture to boiling, add the Tragacanth, and macerate during twenty-four hours, stirring occasionally. Then add enough Water to make the mixture weigh *one hundred grammes*, beat it until it has a uniform consistence, and strain it forcibly through muslin.

Remarks.—This contains 6 per cent. of tragacanth and 18 per cent. of glycerin, and is prepared by adding tragacanth to boiling water mixed with glycerin and straining after maceration for twenty-four hours. The glycerin aids in its preservation. It is a semisolid of much value as a pill excipient, and is practically never used internally.

Dose.—16 Cc. (4 fluidrachms).

MUCILAGO ULMI. Mucilage of Elm.

Recipe.—Elm, bruised, six grammes..... 6 Gm.
Water, one hundred cubic centimeters..... 100 Cc.

Digest the Elm with the Water, on a water-bath, in a covered vessel, during one hour, then strain. This preparation should be freshly made when wanted.

Remarks.—This is made by digesting the 6 per cent. of elm in water during one hour.

Dose.—16 Cc. (4 fluidrachms).

The dose of all the official mucilages is 16 Cc. (4 fluidrachms).

SYRUPS

The next class of preparations to claim our attention are the syrups, which are *preparations containing medicated or flavoring substances combined with a concentrated aqueous solution of sugar.*

A concentrated aqueous solution of sugar is called *syrup* or simple syrup. If the substance added to this is merely a pleasant fruit or an aromatic, the finished product is a *flavored syrup*, while if the addition is of a medicinal character, the product is a *medicated syrup*.

The manufacture of syrups, while it is simple in itself, is rather difficult to explain in a general way by reason of the many modifications in the individual processes which are directed in the pharmacopœia. Moreover, the manufacture of the syrups may be considered from two points of view: first, the method by which the sugar is dissolved, and, second, the method in which the medicinal or flavoring constituent is blended with the sugar.

As far as dissolving the sugar is concerned, this is accomplished in several ways: first, the *solution of sugar with heat*, that is, by placing the sugar in an appropriate vessel, adding water, and possibly a medicated liquid, and warming the mixture until the sugar is dissolved. The second method is by dissolving the sugar in a medicated liquid without the use of heat—the so-called *cold process* of preparing syrups. This process must, in turn, be divided into two methods, the sugar being dissolved in the first case by *agitation*, that is, by simply mixing the sugar with the medicated liquid, and shaking the container from time to time until the sugar is completely dissolved. Also by *percolation*: In this method a piece of sponge or large pledget of absorbent cotton is put in the neck of the percolator, the granulated sugar is poured thereon and packed down as well as circumstances permit, and the requisite amount of the liquid is

poured in on the top. The liquid is attracted downward by the force of gravitation, and in traversing through the sugar dissolves enough of it to make a saturated solution; and in this way, if the requisite quantity of liquid is added, all the sugar will be dissolved, and at the end of the process nothing will remain in the percolator except the straining medium and any insoluble matter that was mixed with the sugar.

For preparing pharmacopœial syrups this process of percolation is permitted, but the writer has found that it possesses the great objection that there is always a considerable quantity of sugar that is not dissolved by the percolating liquid, and in order to redissolve this it is necessary to spread the sugar carefully over the cotton

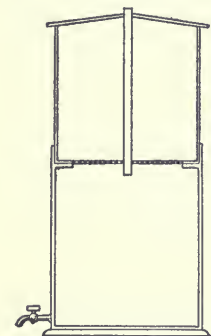


Fig. 163.—Syrup percolator.

plug and pass the liquid through once more. But even this is not always successful.

For the preparation of syrup for soda-fountain use this method of cold percolation is unexcelled, for in this case the complete solution of the sugar is not absolutely necessary. Soda syrup can be prepared in the very simple apparatus shown in Fig. 163. This apparatus was built of two five-gallon castor-oil tins, the lower one provided with a brass faucet, while a sufficient amount of the bottom of the upper one was left to form a margin on which rested a diaphragm of tin, pierced with holes, over which was placed a piece of Canton flannel which served as a strainer. Through the center of this dia-

phragm, up to the top of the upper can, passes a piece of tin tubing intended to permit the exit of air from the lower can. This diaphragm being placed in position, granulated sugar is thrown in the upper can until completely filled, and one gallon of water is poured thereon. The water penetrates the sugar, dissolving it, and is finally passed through the strainer on the diaphragm, dropping into the lower can a saturated solution of sugar and water. When the first gallon of water has passed through the sugar is repacked in the upper can, and a half-gallon of water poured on; and we thus obtain about four to five gallons of a thick, transparent syrup. The sugar remaining in the upper can is poured on paper, dried, and used another time with a fresh quantity of sugar.

As to the special methods of manufacturing syrups, the following table gives a glance at the various processes employed.

TABLE OF OFFICIAL SYRUPS

<i>Process.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparations.</i>
<i>Boiling with sugar.</i>	Syrup of raspberry (U. S. P. 1890).	Syrup of lime.
ADDING MEDICATED LIQUID TO SYRUP.		
MEDICATING LIQUID.		
Aqueous solution.	Syrup of almond. Syrup of citric acid. Syrup of hydriodic acid.	Syrup of ferrous iodide.
Glycerinic solution.	Syrup of phosphates of iron, quinine, and strychnine.	
Fluidextract.	Syrup of krameria. Syrup of rhubarb. Syrup of rubus. Syrup of senega. Syrup of senna.	
Tincture.	Syrup of rhubarb, aromatic.	
Diluted tincture.	Syrup of lactucarium.	
DISSOLVING SUGAR IN THE MEDICATING LIQUID.		
MEDICATING LIQUID.		
Aromatic water.	Syrup of orange flowers.	
Aqueous solution.	Syrup of hypophosphites.	Syrup of calcium lactophosphate.
	Syrup of hypophosphites, compound.	
Mucilage.	Syrup of tar.	
Infusion.	Syrup of acacia.	
Diluted tincture.	Syrup of wild cherry. Syrup of orange. Syrup of tolu.	
	Syrup of ginger.	
Diluted fluidextract.	Syrup of ipecac. Syrup of rose. Syrup of sarsaparilla, compound.	
Vinegar	Syrup of squill, compound. Syrup of squill.	

The table just given affords an insight into the many modifications necessary in preparing the official syrups, and we can now turn to a special consideration of the points regarding the official syrups, giving the same in alphabetic order.

SPECIAL NOTES ON OFFICIAL SYRUPS

SYRUPUS. Syrup.

Recipe.—**Sugar**, in dry crystalline granules, eight hundred and fifty grammes. 850 Gm.
Distilled Water, a sufficient quantity,

To make one thousand cubic centimeters. . . 1000 Cc.

Dissolve the Sugar, with the aid of heat, in *four hundred and fifty cubic centimeters* of Distilled Water, raise the temperature to the boiling-point, strain the liquid, and pass enough Distilled Water through the strainer to make the product, when cold, measure *one thousand cubic centimeters*. Mix thoroughly.

Syrup may also be prepared in the following manner:

Press down into the neck of a percolator of suitable size a pledget of purified cotton, not too tightly, in such a manner that the cotton shall nearly fill the neck of the percolator, and moisten it with a few drops of Distilled Water; introduce the Sugar into the percolator, make its surface level without shaking or jarring, then carefully pour upon it *four hundred and fifty cubic centimeters* of Distilled Water, and regulate the flow of the liquid, if necessary, so that it will pass out in rapid drops. Return the first portions of the percolate until it runs through clear, and when all the liquid has passed, follow it by Distilled Water, added in portions, so that all the Sugar may be dissolved, and the product measure *one thousand cubic centimeters*. Mix thoroughly.

Syrup thus prepared has a specific gravity of about 1.313 at 25° C. (77° F.).

Remarks.—Syrup, or what is commonly called *simple syrup*, is an 85 per cent. solution of sugar in water. It is prepared either by dissolving with boiling water or by percolation of water through sugar. The official syrup should have a specific gravity of about 1.313, and it is exceedingly poor economy to attempt to use a syrup much thinner than this. Syrups containing a smaller quantity of sugar will invariably ferment, and the apparent economy in saving a small quantity of sugar in making the syrup is more than counterbalanced by the necessity of throwing away a batch of spoiled syrup. On the other hand, avoid using more sugar than directed by the pharmacopœia. If the syrup is prepared by percolation, there is no danger of this extra sugar dissolving up; but if the syrup is prepared by warm solution, it will dissolve, and on standing the excess of sugar will crystallize out. As already stated (p. 122), in such cases the portion crystallizing out does not merely represent the excess of sugar, but the moment crystallization has started, the crystals formed will draw from the solution practically all the sugar originally contained therein. The result is that in preparing any of the official syrups the pharmacist using an excess of sugar in order to produce a more suitable preparation defeats the very object sought for, and brings about the very condition which would result were a deficiency of sugar used, for as soon as a large amount of sugar is crystallized out, the remaining mother liquid, being deficient in saccharine strength, commences to ferment. If the syrup is properly prepared, there is no necessity for elaborate precautions of preserving the same, although it should, of course, be kept in well-stoppered bottles.

SYRUPUS ACACIÆ. Syrup of Acacia.

Recipe.—**Acacia**, in selected pieces, one hundred grammes. . . . 100 Gm.
Sugar, eight hundred grammes. 800 Gm.
Distilled Water, four hundred and thirty cubic centimeters. 430 Cc.

To make one thousand cubic centimeters. . . 1000 Cc.

Introduce the Acacia into an enamelled or porcelain dish, add the Distilled Water, and stir occasionally until the Acacia is dissolved; then, having added the Sugar, place the dish on a water-bath and apply heat, gradually increasing the temperature, and stirring from time to time until the Sugar is dissolved. Strain the Syrup, if necessary, and add sufficient Distilled Water to make the product measure *one thousand cubic centimeters*.

Syrup of Acacia should be made in small quantities, and stored in small, tightly stoppered bottles in a cool place.

Remarks.—Syrup of acacia should be freshly prepared. Some pharmacists use it as a pill excipient, although pills so made are usually too hard.

SYRUPUS ACIDI CITRICI. Syrup of Citric Acid.

<i>Recipe.</i> —Citric Acid, <i>ten grammes</i>	10 Gm.
Distilled Water, <i>ten cubic centimeters</i>	10 Cc.
Tincture of Fresh Lemon Peel, <i>ten cubic centimeters</i>	10 Cc.
Syrup, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Dissolve the Citric Acid in the Distilled Water and mix the solution with *five hundred cubic centimeters* of Syrup, then add the Tincture of Fresh Lemon Peel, and lastly enough Syrup to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—This is approximately the average soda-fountain syrup of lemon, although that made from the fresh lemons is much to be preferred. Used entirely as a flavor. No dose given by the pharmacopœia.

SYRUPUS ACIDI HYDRIODICI. Syrup of Hydriodic Acid.

This official derivative of hydriodic acid can best be explained in Part III. (p. 415).

SYRUPUS ALLII (Syrup of Garlic) and SYRUPUS ALTHÆÆ (Syrup of Althæa),

which were recognized by the pharmacopœia of 1890, were dropped in the last revision and are of too little importance to merit more than this passing mention.

SYRUPUS AMYGDALÆ. Syrup of Almond.

<i>Recipe.</i> —Spirit of Bitter Almond, <i>ten cubic centimeters</i>	10 Cc.
Orange Flower Water, <i>one hundred cubic centimeters</i>	100 Cc.
Syrup, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Mix them.

Remarks.—The composition of this syrup underwent a radical change in the last pharmacopœial revision.

Syrup of almond (U. S. P. 1890) was prepared from an emulsion of almond, and was a milky liquid, popular as a flavor in the South under the name of *orgeate syrup*.

Syrup of almond (U. S. P. VIII.), made from spirit of bitter almond, orange flower water, and syrup, is a transparent liquid.

For flavoring purposes it is as satisfactory as the old-time orgeate syrup, and it is much more stable.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS AURANTII. Syrup of Orange.

<i>Recipe</i> .—Tincture of Sweet Orange Peel, <i>fifty cubic centimeters</i>	50 Cc.
Citric Acid, <i>five grammes</i>	5 Gm.
Magnesium Carbonate, <i>ten grammes</i>	10 Gm.
Sugar, <i>eight hundred and twenty grammes</i>	820 Gm.
Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Triturate the Magnesium Carbonate in a mortar with the Tincture, add gradually *four hundred cubic centimeters* of Water, filter, and add sufficient Water through the filter to obtain *four hundred and fifty cubic centimeters* of filtrate; in this dissolve the Citric Acid and Sugar by agitation without heat, and add sufficient Water to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—This is a very palatable flavoring syrup, hence no dose is assigned it in the pharmacopœia.

SYRUPUS AURANTII FLORUM. Syrup of Orange Flowers.

<i>Recipe</i> .—Sugar, <i>eight hundred and fifty grammes</i>	850 Gm.
Orange Flower Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Dissolve the Sugar in *four hundred and fifty cubic centimeters* of Orange Flower Water by agitation, without heat, add enough Orange Flower Water to make the product measure *one thousand cubic centimeters*, and mix thoroughly.

Syrup of Orange Flowers may also be prepared in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour *four hundred and fifty cubic centimeters* of Orange Flower Water upon the Sugar, return the first portions of the percolate until it runs through clear, and, when all the liquid has passed, follow it by Orange Flower Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Another pleasant flavoring syrup.

SYRUPUS CALCII LACTOPHOSPHATIS. Syrup of Calcium Lactophosphate.

This syrup will be considered among the derivatives of lactic acid (p. 514).

SYRUPUS CALCIS. Syrup of Lime.

See p. 507.

SYRUPUS FERRI IODIDI. Syrup of Ferrous Iodide.

Fully described among the compounds of iron (Chapter XXXI).

SYRUPUS FERRI, QUININÆ, ET STRYCHNINÆ PHOSPHATUM.

Syrup of the Phosphates of Iron, Quinine, and Strychnine.

<i>Recipe</i> .—Glycerite of the Phosphates of Iron, Quinine, and Strychnine, <i>two hundred and fifty cubic centimeters</i>	250 Cc.
Syrup, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Mix them. Strain, if necessary.

Remarks.—This official, commonly called *Easton's syrup*, is an excellent tonic, although of rather limited popularity because of its intensely bitter taste. To make a stable preparation is a matter of difficulty, the usual product turning black and eventually precipitating. Hence the use of the glycerite in the present official recipe.

Dose.—4 Cc. (1 fluidrachm), containing about $\frac{1}{80}$ grain of strychnine.

SYRUPUS HYPOPHOSPHITUM. Syrup of Hypophosphites.

<i>Recipe.</i> —Calcium Hypophosphite, <i>forty-five grammes</i>	45 Gm.
Potassium Hypophosphite, <i>fifteen grammes</i>	15 Gm.
Sodium Hypophosphite, <i>fifteen grammes</i>	15 Gm.
Diluted Hypophosphorous Acid, <i>two grammes</i>	2 Gm.
Sugar, <i>six hundred and fifty grammes</i>	650 Gm.
Tincture of Fresh Lemon Peel, <i>five cubic centimeters</i>	5 Cc.
Water, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters*.. 1000 Cc.

Triturate the Hypophosphites with *four hundred and fifty cubic centimeters* of Water, until they are dissolved, add the Tincture of Fresh Lemon Peel, and the Hypophosphorous Acid, and filter the liquid. In the filtrate dissolve the Sugar by agitation, without heat, and add enough water, through the filter, to make the product measure *one thousand cubic centimeters*. Strain, if necessary.

Syrup of Hypophosphites may also be prepared in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour the filtrate obtained as directed in the preceding formula upon the Sugar, return the first portions of the percolate until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—In the manufacture of this syrup, if any difficulty is found in dissolving the calcium hypophosphite, the fault is apt to lie in the salt, as the large amount of commercial calcium hypophosphite is not pure.

Dose.—8 Cc. (2 fluidrachms).

SYRUPUS HYPOPHOSPHITUM COMPOSITUS. Compound Syrup of Hypophosphites.

<i>Recipe.</i> —Calcium Hypophosphite, <i>thirty-five grammes</i>	35.00 Gm.
Potassium Hypophosphite, <i>seventeen and one-half grammes</i>	17.50 Gm.
Sodium Hypophosphite, <i>seventeen and one-half grammes</i>	17.50 Gm.
Ferric Hypophosphite, <i>two and one-fourth grammes</i>	2.25 Gm.
Manganese Hypophosphite, <i>two and one-fourth grammes</i>	2.25 Gm.
Quinine, <i>one and one-tenth grammes</i>	1.10 Gm.
Strychnine, <i>one hundred and fifteen milligrammes</i>	0.115 Gm.
Sodium Citrate, <i>three and three-fourths grammes</i> ..	3.75 Gm.
Diluted Hypophosphorous Acid, <i>fifteen cubic centimeters</i>	15.00 Cc.
Sugar, <i>seven hundred and seventy-five grammes</i> ...	775.00 Gm.
Water, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters* 1000.00 Cc.

Rub the Ferric and Manganese Hypophosphites with the Sodium Citrate, add *thirty cubic centimeters* of Water, and warm the mixture for a few minutes, until a

clear greenish solution is obtained. Dissolve the Calcium, Potassium, and Sodium Hypophosphites in *four hundred and fifty cubic centimeters* of Water, to which *five cubic centimeters* of Diluted Hypophosphorous Acid has previously been added; then dissolve the Quinine and Strychnine in *thirty cubic centimeters* of Water with the aid of *ten cubic centimeters* of Diluted Hypophosphorous Acid, and finally dissolve the Sugar, with agitation, in these solutions, previously mixed.

Strain the Syrup, if necessary, and add sufficient Water, through the strainer, to make the product measure *one thousand cubic centimeters*.

Remarks.—This new official, which is practically identical with the preparation of the same name of the former National Formulary, is intended to replace the many commercial varieties of syrup of hypophosphites. For the first time we have official a syrup of hypophosphites designated as “compound,” something that was much desired, since formerly a pharmacist receiving a prescription calling for “Syr. Hypophos. Co.” was in a quandary as to whether the physician desired Syrup of Hypophosphites, U. S. P., Syrup of Hypophosphites with Iron, U. S. P. 1890, Syrup of Hypophosphites, N. F., or one of the many proprietary syrups bearing that name.

Dose.—8 Cc. (2 fluidrachms).

Syrup of hypophosphites with iron (U. S. P. 1890) was the official syrup of hypophosphites combined with ferrous lactate. It was never very popular, the present official compound syrup of hypophosphites being a much more satisfactory product.

SYRUPUS IPECACUANHÆ. Syrup of Ipecac.

<i>Recipe.</i> —Fluidextract of Ipecac, <i>seventy cubic centimeters</i> . . .	70 Cc.
Acetic Acid, <i>ten cubic centimeters</i>	10 Cc.
Glycerin, <i>one hundred cubic centimeters</i>	100 Cc.
Sugar, <i>seven hundred grammes</i>	700 Gm.
Water, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters* . . . 1000 Cc.

Dilute the Fluidextract of Ipecac with *three hundred cubic centimeters* of Water to which the Acetic Acid has previously been added, and mix them thoroughly by shaking; set the liquid aside in a cool place for twenty-four hours. Then filter, and pass enough Water through the filter to obtain *four hundred and fifty cubic centimeters* of filtrate. To this liquid add the Glycerin, dissolve the Sugar in the mixture, and add enough Water to make the product measure *one thousand cubic centimeters*. Mix thoroughly and strain, if necessary.

Syrup of Ipecac may also be prepared in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*.

Mix the filtrate obtained as directed in the preceding formula with the Glycerin, pour the mixture upon the Sugar, return the first portions of the percolate, until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—The addition of acetic acid is to insure solution of the alkaloid emetine.

Dose.—Expectorant, 1 Cc. (15 minims). Emetic, 15 Cc. (4 fluidrachms).

SYRUPUS KRAMERIÆ. Syrup of Krameria.

<i>Recipe.</i> —Fluidextract of Krameria, <i>four hundred and fifty cubic centimeters</i>	450 Cc.
Syrup, <i>five hundred and fifty cubic centimeters</i>	550 Cc.

To make *one thousand cubic centimeters* . . 1000 Cc.

Mix them.

Remarks.—Note that while ten syrups (*krameria*, *rhubarb*, *rubus*, *senega*, *senna*, *ginger*, *ipecac*, *rose*, *sarsaparilla* compound, and *squill* compound) are made from the fluidextract, the first five are made by simply mixing the fluidextract with the syrup, while in making the last five the fluidextract is diluted with water and other ingredients, filtered, and sugar dissolved in the perfectly transparent filtrate. The latter process—shown in the recipe for syrup of *ipecac* given just above—is much preferable merely to mixing the fluidextract with syrups (as in making syrup of *krameria*), which almost invariably yields a cloudy, unsightly product.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS LACTUCARII. Syrup of Lactucarium.

<i>Recipe.</i> —Tincture of Lactucarium, one hundred cubic centimeters.....	100 Cc.
Glycerin, two hundred cubic centimeters.....	200 Cc.
Citric Acid, one gramme.....	1 Gm.
Orange Flower Water, fifty cubic centimeters.....	50 Cc.
Syrup, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Tincture of Lactucarium with the Glycerin, add the Orange Flower Water in which the Citric Acid has been previously dissolved, and filter, if necessary. Finally, add a sufficient quantity of Syrup to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Dose.—8 Cc. (2 fluidrachms).

SYRUPUS PICIS LIQUIDÆ. Syrup of Tar.

<i>Recipe.</i> —Tar, five grammes.....	5 Gm.
Alcohol, fifty cubic centimeters.....	50 Cc.
Magnesium Carbonate, ten grammes.....	10 Gm.
Sugar, eight hundred and fifty grammes.....	850 Gm.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Tar intimately, in a mortar, with *ten grammes* of clean white sand, add *one hundred cubic centimeters* of Water, and, after kneading the mass thoroughly with the pestle, pour off the Water and throw it away. Treat the residue with the Alcohol, and, when the Tar is dissolved, add the Magnesium Carbonate and *fifty grammes* of Sugar, and after thorough trituration add *four hundred cubic centimeters* of Water; stir the mixture occasionally during two hours, and filter. Dissolve the remainder of the Sugar in the clear filtrate by gentle heat, strain, and add sufficient Water to make the product measure *one thousand cubic centimeters*.

Remarks.—In making this syrup the washed tar is dissolved in alcohol, triturated with magnesium carbonate and water, and then filtered. In the resulting tar water the sugar is dissolved. For general purposes, as in cough syrups, a syrup prepared from glycerole of tar (p. 847) (1 drachm glycerole to the fluidounce) is more convenient.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS PRUNI VIRGINIANÆ. Syrup of Wild Cherry.

<i>Recipe.</i> —Wild Cherry, in No. 20 powder, one hundred and fifty grammes.....	150 Gm.
Sugar, seven hundred grammes.....	700 Gm.
Glycerin, one hundred and fifty cubic centimeters.....	150 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Moisten the Wild Cherry with a sufficient quantity of Water, and macerate for twenty-four hours in a covered vessel; introduce the Glycerin into a graduated receiving bottle; pack the Wild Cherry firmly in a cylindrical percolator, and gradually pour Water upon it; continue the percolation (shaking the percolate occasionally with the Glycerin), until the liquid measures *four hundred and fifty cubic centimeters*. Dissolve the Sugar in the liquid by agitation, without heat, strain, and pass enough Water through the strainer to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Syrup of Wild Cherry may also be prepared in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour the percolate obtained as directed in the preceding formula upon the Sugar, return the first portions of the percolate, until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—This syrup, which enjoys just popularity, is made by dissolving sugar in a cold percolated infusion of the bark previously mixed with glycerin. Not the official infusion, however, but a much stronger product.

In the former pharmacopœia the glycerin was mixed with the water before percolating the bark—that is, it was a part of the menstruum. This plan was dropped in the last revision, since it is claimed that the glycerin dissolves the tannin of the bark, giving a bitter syrup. Whether this change is an improvement is still a matter of controversy.

It is imperative that heat be avoided in making this syrup, since heat prevents the formation or causes the volatilization of the active principle, hydrocyanic acid. (See p. 778.)

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS RHEI. Syrup of Rhubarb.

<i>Recipe.</i> —Fluidextract of Rhubarb, <i>one hundred cubic centimeters</i>	100 Cc.
Spirit of Cinnamon, <i>four cubic centimeters</i>	4 Cc.
Potassium Carbonate, <i>ten grammes</i>	10 Gm.
Water, <i>fifty cubic centimeters</i>	50 Cc.
Syrup, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters*.. 1000 Cc.

Mix the Spirit of Cinnamon with the Fluidextract of Rhubarb, and add to it the Potassium Carbonate previously dissolved in the Water, and lastly enough Syrup to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Syrup of rhubarb is prepared from the fluidextract of rhubarb and spirit of cinnamon, potassium carbonate being employed to aid in dissolving the difficultly soluble resins found in the rhubarb. An aqueous solution is thus obtained, in which the requisite amount of sugar is dissolved. This syrup is a laxative, while *aromatic syrup of rhubarb* is chiefly astringent, the aromatic it contains bringing about this different action. More concerning this therapeutic difference will be said when the drug, rhubarb, is discussed.

Dose.—8 Cc. (2 fluidrachms).

SYRUPUS RHEI AROMATICUS. Aromatic Syrup of Rhubarb.

<i>Recipe.</i> —Aromatic Tincture of Rhubarb, <i>one hundred and fifty cubic centimeters</i>	150 Cc.
Potassium Carbonate, <i>one gramme</i>	1 Gm.
Syrup, <i>eight hundred and fifty cubic centimeters</i>	850 Cc.
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Dissolve the Potassium Carbonate in the Aromatic Tincture of Rhubarb. Filter, if necessary, and add sufficient Syrup to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—This syrup is made by mixing syrup with aromatic tincture of rhubarb, previously rendered alkaline with potassium carbonate. The alkali is employed for the same reason as in making syrup of rhubarb. Turn into aromatic tincture of rhubarb (p. 235), and note what aromatics it contains.

Dose.—8 Cc. (2 fluidrachms).

SYRUPUS ROSÆ. Syrup of Rose.

<i>Recipe.</i> —Fluidextract of Rose, one hundred and twenty-five cubic centimeters.....	125 Cc.
Diluted Sulphuric Acid, ten cubic centimeters.....	10 Cc.
Sugar, seven hundred and fifty grammes.....	750 Gm.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Fluidextract of Rose and Diluted Sulphuric Acid with *three hundred cubic centimeters* of Water; after allowing the mixture to stand two hours, filter, and dissolve the Sugar in the clear filtrate, by agitation. Finally, add a sufficient quantity of Water to make the product measure *one thousand cubic centimeters*. Strain, if necessary.

Remarks.—In making this syrup the fluidextract is diluted with water, diluted sulphuric acid being added to facilitate solution, and sugar dissolved in the clear filtrate. The result is a product much more handsome than that made by the process of 1890, when the fluidextract was merely mixed with syrup, producing a turbid liquid. The syrup is used entirely as a flavor, hence no dose is given by the pharmacopœia.

SYRUPUS RUBI. Syrup of Rubus.

<i>Recipe.</i> —Fluidextract of Rubus, two hundred and fifty cubic centimeters.....	250 Cc.
Syrup, seven hundred and fifty cubic centimeters....	750 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them.

Remarks.—An unsightly liquid, made by mixing the fluidextract with syrup.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS RUBI IDÆI (U. S. P. 1890). Syrup of Raspberry.

Syrup of raspberry is of greater interest at the soda fountain than behind the prescription counter. The pharmacopœia of 1890 directed that this syrup be made from the fresh fruit, not from commercial fruit-juices.

SYRUPUS SARSAPARILLÆ COMPOSITUS. Compound Syrup of Sarsaparilla.

<i>Recipe.</i> —Fluidextract of Sarsaparilla, two hundred cubic centimeters.....	200.0 Cc.
Fluidextract of Glycyrrhiza, fifteen cubic centimeters.....	15.0 Cc.
Fluidextract of Senna, fifteen cubic centimeters....	15.0 Cc.
Sugar, six hundred and fifty grammes.....	650.0 Gm.
Oil of Sassafras, two-tenths of a cubic centimeter....	0.2 Cc.
Oil of Anise, two-tenths of a cubic centimeter.....	0.2 Cc.
Oil of Gaultheria, two-tenths of a cubic centimeter....	0.2 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000.0 Cc.

Add the Oils (equivalent to about *four drops* each) to the mixed Fluidextracts and shake the liquid thoroughly. Then add enough Water to make up the volume to *six hundred cubic centimeters*, and mix well. Set the mixture aside for one hour, and then filter it. Dissolve the Sugar in the filtrate with the aid of a gentle heat, allow the liquid to cool, strain, and add enough Water, through the strainer, to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Compound syrup of sarsaparilla affords a good test of memory, and its constituents form a question of which examining boards are quite fond. Note the three fluidextracts and the three oils used in its manufacture. The uses of the syrup as an adjuvant, especially for quinine, are well known.

Dose.—16 Cc. (4 fluidrachms).

SYRUPUS SCILLÆ. Syrup of Squill.

<i>Recipe.</i> —Vinegar of Squill, four hundred and fifty cubic centimeters.....	450 Cc.
Sugar, eight hundred grammes.....	800 Gm.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Sugar in the Vinegar of Squill with the aid of a gentle heat, then strain, and, when the strained liquid is cold, add enough Water, through the strainer, to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Syrup of squill is now made by dissolving sugar in vinegar of squill at gentle heat. In the pharmacopœia of 1890 the vinegar of squill was first boiled and filtered; this is not now necessary, since the boiling is performed in the manufacture of the official vinegar. (See p. 263.)

Dose.—2 Cc. (30 minims).

SYRUPUS SCILLÆ COMPOSITUS. Compound Syrup of Squill.

<i>Recipe.</i> —Fluidextract of Squill, eighty cubic centimeters....	80 Cc.
Fluidextract of Senega, eighty cubic centimeters....	80 Cc.
Antimony and Potassium Tartrate, two grammes..	2 Gm.
Purified Talc, twenty grammes.....	20 Gm.
Sugar, seven hundred and fifty grammes.....	750 Gm.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Fluidextracts, evaporate them, in a tared dish, on a water-bath, to *one hundred grammes*, and mix the residue with *three hundred and fifty cubic centimeters*

of Water. When the mixture is cold, incorporate with it, intimately, the Purified Talc, filter, pass enough Water through the filter to obtain *four hundred cubic centimeters* of clear filtrate, and add to this the Antimony and Potassium Tartrate previously dissolved in *twenty-five cubic centimeters* of hot Water. Dissolve the Sugar in this liquid by agitation, without heat, strain, and add enough Water, through the strainer, to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Compound Syrup of Squill may also be prepared in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour the filtrate obtained as directed in the preceding formula, and mixed with the solution of Antimony and Potassium Tartrate, upon the Sugar, return the first portions of the percolate, until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Compound spirit of squill is made from the evaporated fluidextracts of squill and senega, the aqueous solution of the active principles of these drugs being filtered through purified talc, and to the clear filtrate a solution of tartar emetic is added, and in the resultant liquid sugar is dissolved. It is called *Coxe's hive syrup*.

Dose.—2 Cc. (30 minims).

SYRUPUS SENEGÆ. Syrup of Senega.

<i>Recipe.</i> —Fluidextract of Senega, two hundred cubic centimeters.....	200 Cc.
Syrup, eight hundred cubic centimeters.....	800 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them.

Remarks.—The recipe of the present pharmacopœia is markedly different from that of the pharmacopœia of 1890. The syrup of 1890 was made by diluting the fluidextract with water, to which was added ammonia water, to dissolve the pectin found in the senega, filtering, and dissolving the sugar in the filtrate, while the syrup of the present pharmacopœia is made by mixing the fluidextract with syrup. The fluidextract now official is, however, prepared with alkali (see p. 250), which holds the pectin in solution.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS SENNÆ. Syrup of Senna.

<i>Recipe.</i> —Fluidextract of Senna, two hundred and fifty cubic centimeters.....	250 Cc.
Oil of Coriander, five cubic centimeters.....	5 Cc.
Syrup, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Oil of Coriander with the Fluidextract of Senna and add a sufficient quantity of Syrup to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—The above recipe differs widely from that of the pharmacopœia of 1890, which called for an infusion of senna to which was added oil of coriander dissolved in alcohol, the mixture filtered, and sugar dissolved in the filtrate.

Dose.—4 Cc. (1 fluidrachm).

SYRUPUS TOLUTANUS. Syrup of Tolu.

<i>Recipe.</i> —Tincture of Tolu, <i>fifty cubic centimeters</i>	50 Cc.
Magnesium Carbonate, <i>ten grammes</i>	10 Gm.
Sugar, <i>eight hundred and twenty grammes</i>	820 Gm.
Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Rub the Tincture of Tolu, in a mortar, with the Magnesium Carbonate and *sixty grammes* of the Sugar. Then gradually add *four hundred and fifty cubic centimeters* of Water, with constant trituration, and filter. Dissolve the remainder of the Sugar in the clear filtrate, with the aid of a gentle heat, strain the Syrup while hot, and add a sufficient quantity of Water to make the product measure *one thousand cubic centimeters*.

Syrup of Tolu may also be made in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour the filtrate obtained as directed in the preceding formula upon the Sugar, return the first portions of the percolate, until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—The official recipe just given is practically a return to the process of the pharmacopœia of 1870, and yields a handsome aromatic yellow syrup. It is an interesting example of reversion to a tried old recipe after two decades of experiment. The process of 1880—digesting a mixture of balsam of tolu and sugar with water—was cumbersome and yielded a poor product, and the process of 1890—dissolving sugar in a water of tolu made by rubbing an alcoholic solution of the balsam with precipitated calcium phosphate and water—was not much better.

Dose.—16 Cc. (4 fluidrachms).

SYRUPUS ZINGIBERIS. Syrup of Ginger.

<i>Recipe.</i> —Fluidextract of Ginger, <i>thirty cubic centimeters</i>	30 Cc.
Alcohol, <i>twenty cubic centimeters</i>	20 Cc.
Magnesium Carbonate, <i>ten grammes</i>	10 Gm.
Sugar, <i>eight hundred and twenty grammes</i>	820 Gm.
Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Mix the Fluidextract of Ginger and the Alcohol, then triturate the liquid in a mortar with the Magnesium Carbonate and *sixty grammes* of the Sugar. Then gradually add *four hundred and fifty cubic centimeters* of Water, with constant trituration, and filter. Dissolve the remainder of the Sugar in the clear filtrate, with the aid of a gentle heat, strain the Syrup while hot, and add a sufficient quantity of Water to make the product measure *one thousand cubic centimeters*.

Syrup of Ginger may also be made in the following manner:

Prepare a percolator or funnel in the manner described under *Syrupus*. Pour the filtrate obtained as directed in the preceding formula upon the Sugar, return the first portions of the percolate, until it runs through clear, and, when all the liquid has passed, follow it with Water, until the product measures *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—Syrup of ginger is prepared like syrup of tolu, except that it is the fluidextract that is rubbed with magnesium carbonate and water and not the tincture.

Dose.—16 Cc. (4 fluidrachms).

DOSES OF OFFICIAL SYRUPS

1 Cc. (15 minims).....	Syrups of ferrous iodide and of ipecac (as an expectorant).
2 Cc. (30 minims).....	Syrups of lime, squill, and squill compound.
4 Cc. (1 fluidrachm).....	Syrups of almond, hydriodic acid, krameria, phosphates of iron, quinine and strychnine, rubus, senega, senna, tar, and wild cherry.
8 Cc. (2 fluidrachms).....	Syrups of calcium lactophosphate, hypophosphites compound, lactucarium, rhubarb, and rhubarb aromatic.
16 Cc. (4 fluidrachms).....	Syrups of ginger, sarsaparilla compound, and tolu.

No dose given the official syrups not named above, as all the rest are flavored syrups and of no medicinal action.

HONEYS

Honeys are *liquids containing medicines blended with honey*. In ancient times the honeys represented the most popular class of medicinal preparations, but in our present day of elixirs and of syrups the honeys are falling into disuse, although one is still recognized by the pharmacopœia. Honey itself is the natural product stored in the honeycombs by the bees, as explained on p. 705.

MEL DEPURATUM. Clarified Honey.

Recipe.—**Honey**, a convenient quantity.
Distilled Water,
Glycerin, each, a sufficient quantity.

Mix the Honey intimately with *two per cent.* of its weight of paper-pulp, which has been previously reduced to shreds, thoroughly washed and soaked in water, and then strongly expressed and again shredded. Then apply the heat of a water-bath, and, as long as any scum rises to the surface, carefully remove this. Finally, add enough Distilled Water to make up the loss incurred by evaporation, strain, and mix the strained liquid with *five per cent.* of its weight of glycerin.

Clarified Honey should conform to the tests of purity given under *Mel*.

Remarks.—Clarified honey—*Mel Despumatum* (U. S. P. 1890)—is natural honey, clarified by mixing with shreds of wetted filter-paper and heating on a water-bath, carefully removing the scum and filter-paper as it rises to the surface (p. 136). When the clarification is complete, the liquid is strained and mixed with 5 per cent. of glycerin in order to preserve it.

Dose.—4 Cc. (1 fluidrachm).

MEL ROSÆ. Honey of Rose.

Recipe.—**Fluidextract of Rose**, one hundred and twenty cubic centimeters..... 120 Cc.
Clarified Honey, a sufficient quantity,
To make one thousand grammes.. 1000 Gm.

Into a tared vessel introduce the Fluidextract of Rose, then add enough Clarified Honey to make the contents weigh *one thousand grammes*, and mix thoroughly.

Remarks.—Honey of rose is prepared by blending 12 per cent. of fluidextract of rose with clarified honey. Honey of rose is used in preparation of *mass of mercury* (p. 311).

Dose.—4 Cc. (1 fluidrachm).

Doses of Honeys.—All three official honeys—honey, clarified honey, and honey of rose—are directed by the pharmacopœia in doses of 4 Cc. (1 fluidrachm). That no evil effect can result from this dose goes without saying.

CHAPTER XIII

ELIXIRS, GLYCERITES, COLLODIONS, AND OLEATES

ELIXIRS

ELIXIRS are *alcoholic solutions of medicinal substances containing sugar*. Therefore, it can be seen that there is a slight analogy between them and syrups. In the latter, however, there is much more sugar. Considering the extensive use of elixirs, it may seem strange that so few have been introduced into the pharmacopœia, but three (adjuvant elixir, aromatic elixir, and elixir of iron, quinine, and strychnine phosphates) being official.

There are two reasons for this. The first is that elixirs are not regarded as preparations whose popularity will stand the test of time. Already it is noticed that they are not used as much as they were five years ago; therefore, the pharmacopœial committee thought it unwise to introduce a long line of preparations in 1900 only to have them dismissed in 1910. The second reason is that the comparatively large doses of these pleasant alcoholic preparations cause fear that they are more to be regarded as tipples than as true medicinal agents. Therefore, the pharmacopœial committee omitted the long line of these elegant pharmaceuticals, leaving them for incorporation in the National Formulary, and in that valuable work a full list of the more popular elixirs will be found.

But another word concerning elixirs; do not fear that they will spoil. If properly made, they improve with age, for time enables the alcohol and aromatics to blend, making a smoother, more pleasant preparation.

SPECIAL NOTES ON OFFICIAL ELIXIRS

ELIXIR ADJUVANS. Adjuvant Elixir.

<i>Recipe.</i> —Fluidextract of Glycyrrhiza, one hundred and twenty cubic centimeters.....	120 Cc.
Aromatic Elixir, eight hundred and eighty cubic centimeters.....	880 Cc.
To make one thousand cubic centimeters..	1000 Cc.
Mix, and filter if necessary.	

Remarks.—This new official has been taken from the old National Formulary, and, as the name indicates, is intended as a pleasant addition to mixtures of bitter drugs, the glycyrrhiza being an admirable mask for bitter substances. No dose is given by the pharmacopœia.

ELIXIR AROMATICUM. Aromatic Elixir.

Recipe.—Compound Spirit of Orange, *twelve cubic centimeters*..... 12 Cc.
 Syrup, *three hundred and seventy-five cubic centimeters*..... 375 Cc.
 Purified Talc, *thirty grammes*..... 30 Gm.
 Alcohol,
 Distilled Water, each, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

To the Compound Spirit of Orange, add enough Alcohol to make *two hundred and fifty cubic centimeters*. To this solution, add the Syrup in several portions, agitating after each addition, and afterward add, in the same manner, *three hundred and seventy-five cubic centimeters* of Distilled Water. Mix the Purified Talc intimately with the liquid, and then filter through a wetted filter, returning the first portions of the filtrate until a transparent liquid is obtained. Lastly, wash the filter with a mixture of *one volume* of Alcohol and *three volumes* of Distilled Water, until the product measures *one thousand cubic centimeters*.

Remarks.—This is a pleasant flavored preparation, provided the compound spirit of orange (p. 178) is made from fine oils. It is the basis of most elixirs, among them the official *adjuvant elixir* and *elixir of iron, quinine, and strychnine phosphates*.

ELIXIR FERRI, QUININÆ ET STRYCHNINÆ PHOSPHATUM.
Elixir of Iron, Quinine, and Strychnine Phosphates.

Recipe.—Soluble Ferric Phosphate, *seventeen and one-half grammes*..... 17.500 Gm.
 Quinine, *eight and seventy-five hundredths grammes*..... 8.750 Gm.
 Strychnine, *two hundred and seventy-five milligrammes*..... 0.275 Gm.
 Phosphoric Acid, *two cubic centimeters*..... 2.000 Cc.
 Ammonium Carbonate, in translucent pieces, *nine grammes*..... 9.000 Gm.
 Alcohol, *sixty cubic centimeters*..... 60.000 Cc.
 Acetic Acid, *twenty-eight and sixty-five hundredth grammes*..... 28.650 Gm.
 Ammonia Water,
 Distilled Water,
 Aromatic Elixir, each, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000.000 Cc.

Dissolve the Quinine and Strychnine in the Alcohol, then add the Phosphoric Acid and *three hundred and fifty cubic centimeters* of Aromatic Elixir. Add the Acetic Acid to the Ammonium Carbonate, contained in a suitable vessel, and when solution is complete, neutralize with Ammonia Water and add enough Distilled Water to make the product measure *fifty cubic centimeters*. Mix the solution of Ammonium Acetate with the solution of the alkaloids, and add enough Aromatic Elixir to make the liquid measure *eight hundred and eighty cubic centimeters*. Dissolve the Ferric Phosphate in *thirty cubic centimeters* of Distilled Water, by the aid of a gentle heat, and if the solution be acid to litmus-paper, neutralize exactly with Ammonia Water, and add enough Aromatic Elixir to make the product measure *one hundred and twenty cubic centimeters*. Finally, mix the two solutions and filter if necessary.

Remarks.—The manufacture of this preparation requires considerable care in order to secure a handsome amber liquid. It is similar to the syrup of the phosphates of iron, quinine, and strychnine, but contains a trifle more strychnine.

Dose.—4 Cc. (1 fluidrachm), containing about $\frac{1}{60}$ grain strychnine.

ELIXIR PHOSPHORI (U. S. P. 1890). Elixir of Phosphorus.

Elixir of phosphorus of the former pharmacopœia was made by mixing 210 Cc. spirit of phosphorus (U. S. P. 1890), 2 Cc. oil of anise, 550 Cc. glycerin, and aromatic elixir to make 1000 Cc. Each cubic centimeter contained about $\frac{1}{4}$ milligramme of phosphorus, making the average dose (4 Cc.) contain 1 milligramme ($\frac{1}{80}$ grain) of phosphorus.

GLYCERITES

Glycerites (or *glycerols*) are *mixtures or solutions of medicinal substances with glycerin*. Most of them are solutions, but one of those official (glycerite of starch) is a semisolid mass. The glycerites are excellent preparations, being both permanent and emollient.

TABLE OF OFFICIAL GLYCERITES

<i>Process.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparation.</i>
<i>Cold solution.</i>	Glycerite of the phosphates of iron, quinine, and strychnine. Glycerite of phenol.	
<i>Warm solution.</i>	Glycerite of tannic acid. Glycerite of starch.	Glycerite of boroglycerin.
<i>Percolation and solution.</i>	Glycerite of hydrastis.	

SPECIAL NOTES ON OFFICIAL GLYCERITES**GLYCERITUM ACIDI TANNICI. Glycerite of Tannic Acid.**

<i>Recipe.</i> —Tannic Acid, twenty grammes.....	20 Gm.
Glycerin, eighty grammes.....	80 Gm.
To make one hundred grammes	100 Gm.

Triturate the Tannic Acid with the Glycerin to a smooth paste, transfer this to a porcelain dish, avoiding contact with metallic utensils, and apply the heat of a water-bath, until the Tannic Acid is completely dissolved. Then transfer the solution to a bottle.

Remarks.—Glycerite of tannic acid contains 20 per cent. of tannic acid, and is made by warming the mixture of tannic acid on a water-bath until the acid is completely dissolved. It is hardly necessary to say that the use of metallic utensils should be rigidly avoided, as tannin combines with metals, particularly with iron, forming with that metal an inky black compound.

The glycerite when first prepared is of a brownish color, but on standing it assumes a deep green tint.

It is a convenient form for dispensing solutions of tannic acid, it being readily diluted by the addition of as much water as necessary.

Dose.—2 Cc. (30 minims).

GLYCERITUM AMYLI. Glycerite of Starch.

<i>Recipe.</i> —Starch, ten grammes.....	10 Gm.
Water, ten cubic centimeters.....	10 Cc.
Glycerin, eighty grammes.....	80 Gm.

Triturate the Starch with the Water, until a homogeneous mixture is produced. Then gradually add this to the Glycerin, contained in a porcelain dish, and heated to about 140° C. (284° F.). Continue the heat, with constant stirring, keeping it below 144° C. (291.2° F.), until a translucent jelly is formed. Transfer the product to suitable vessels, provided with well-fitting covers.

Glycerite of starch is a jelly containing 10 per cent. of starch, made by blending starch, water, and glycerin, and gently warming, with constant stirring, on a Bunsen burner. The stirring is necessary to avoid scorching.

Glycerite of starch is used as a basis of enemas and also as a pill excipient. Not being used internally, no dose is given by the pharmacopœia.

GLYCERITUM BOROGLYCERINI. Glycerite of Boroglycerin.

Recipe.—Boric Acid, in fine powder, three hundred and ten grammes..... 310 Gm.
Glycerin, a sufficient quantity,
To make one thousand grammes.. 1000 Gm.

Heat four hundred and sixty grammes of Glycerin, in a tared porcelain dish, to a temperature not exceeding 150° C. (302° F.), and add the Boric Acid in portions, constantly stirring. When all is added and dissolved, continue the heat at the same temperature, frequently stirring, and breaking up the film which forms on the surface. When the mixture has been reduced to the weight of five hundred grammes, add to it five hundred grammes of Glycerin, mix thoroughly, and transfer it to suitable vessels.

Remarks.—Glycerite of boroglycerin contains 50 per cent. of boroglycerin (for calculations, see p. 682), and is a thick, viscid liquid, which is used externally as an antiseptic and emollient dressing.

GLYCERITUM FERRI, QUININÆ, ET STRYCHNINÆ PHOSPHATUM. Glycerite of the Phosphates of Iron, Quinine, and Strychnine.

Recipe.—Soluble Ferric Phosphate, eighty grammes..... 80.0 Gm.
Quinine, one hundred and four grammes..... 104.0 Gm.
Strychnine, eight-tenths of a gramme..... 0.8 Gm.
Phosphoric Acid, two hundred cubic centimeters..... 200.0 Cc.
Glycerin, five hundred cubic centimeters..... 500.0 Cc.
Water, a sufficient quantity,
To make one thousand cubic centimeters.. 1000.0 Cc.

Heat the Soluble Ferric Phosphate with two hundred cubic centimeters of Water, in a porcelain dish, at a temperature not exceeding 70° C. (158° F.), until it is dissolved. Then add the Phosphoric Acid with the Strychnine and Quinine, and sufficient Water to make the product measure five hundred cubic centimeters, and stir until solution is effected. Mix the solution with the Glycerin, and filter if necessary.

Remarks.—This new official is used almost exclusively for the manufacture of Easton's syrup (p. 682).

Dose.—1 Cc. (15 minims).

GLYCERITUM HYDRASTIS. Glycerite of Hydrastis.

Recipe.—Hydrastis, in No. 60 powder, one thousand grammes. 1000 Gm.
Glycerin, five hundred cubic centimeters..... 500 Cc.
Alcohol,
Water, each, a sufficient quantity,
To make one thousand cubic centimeters.. 1000 Cc.

Moisten the Hydrastis with three hundred and fifty cubic centimeters of Alcohol, and pack it firmly in a cylindrical percolator; then add enough Alcohol to saturate the

powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Alcohol, until the Hydrastis is practically exhausted. Remove nearly all of the Alcohol by distillation or evaporation, pour the thick concentrated liquid into *five hundred cubic centimeters* of ice-cold Water, and set it aside, in a cold place, for twenty-four hours. Then filter, pass enough cold Water through the filter to make the filtrate measure *five hundred cubic centimeters*, add the Glycerin, and mix thoroughly.

Average Dose.—2 Cc. (30 minims).

Remarks.—Glycerite of hydrastis is practically a glycerinic fluid-extract of that important drug. It is prepared by percolating the drug with alcohol, evaporating off the alcohol, and blending the filtered residue with glycerin. It represents a form of “fluid hydrastis” so largely used as an ingredient of gonorrheal injections.

Dose.—2 Cc. (30 minims).

GLYCERITUM PHENOLIS. Glycerite of Phenol.

<i>Recipe.</i> —Liquefied Phenol, <i>twenty cubic centimeters</i>	20 Cc.
Glycerin, <i>eighty cubic centimeters</i>	80 Cc.
To make <i>one hundred cubic centimeters</i> ..	100 Cc.

Add the Liquefied Phenol to the Glycerin, and stir until thoroughly mixed.

Remarks.—Glycerite of carbolic acid, as this preparation was called by the pharmacopœia of 1890, and as it is commonly called, contains 20 per cent. of liquefied phenol, and is an admirable antiseptic; for this purpose, however, it is a trifle too powerful, and more advantageous is the so-called *carbulated glycerin*, containing one fluidrachm of carbolic acid to the fluidounce of the finished product.

Dose.—0.3 Cc. (5 minims).

GLYCERITUM VITELLI (U. S. P. 1890). Glycerite of Yolk of Egg.

Glycerite of yolk of eggs of the former pharmacopœia or *glyconin* was made by rubbing up 45 Gm. of yolk of egg with 55 Gm. of glycerin. A thick yellow mixture results, which affords one of the most striking illustrations of the antiseptic action of glycerin. The writer has in his possession a sample glycerite of the yolk of egg prepared five years ago, and which is as sweet as when first made.

DOSES OF GLYCERITES

0.3 Cc. (5 minims).....	Glycerite of phenol.
1 Cc. (15 minims).....	Glycerite of phosphate of iron, quinine, and strychnine.
2 Cc. (30 minims).....	Glycerite of hydrastis and of tannic acid.

No dose given of glycerite of boroglycerin nor of glycerite of starch.

COLLODIONS

These are *preparations used externally, having as their base collodion*, which liquid is a solution of pyroxylin (gun-cotton) in alcohol and ether (p. 702).

The solvent readily evaporates, leaving a film of gun-cotton, which is an excellent protective.

TABLE OF OFFICIAL COLLODIONS

(ALL GALENIC)

<i>Made by simple solution</i>	{ Collodion, Flexible collodion. Styptic collodion.
<i>Made by percolation and solution</i> ..	Cantharidal collodion.

SPECIAL NOTES ON OFFICIAL COLLODIONS

COLLODIUM. Collodion.

<i>Recipe</i> .—Pyroxylin, forty grammes.....	40 Gm.
Ether, seven hundred and fifty cubic centimeters....	750 Cc.
Alcohol, two hundred and fifty cubic centimeters....	250 Cc.
To make about one thousand cubic centimeters..	1000 Cc.

To the Pyroxylin, contained in a suitable bottle, add the Ether, and allow it to stand for fifteen minutes; then add the Alcohol, and shake the bottle until the Pyroxylin is dissolved. Cork the bottle well, and set it aside until the liquid has become clear. Finally, decant the clear portion from any sediment which may have deposited, and transfer it to bottles, which should be well corked and sealed.

Keep the Collodion in a cool place, remote from lights or fire.

Remarks.—This ethereal solution of gun-cotton will be discussed later (p. 702).

COLLODIUM CANTHARIDATUM. Cantharidal Collodion.

<i>Recipe</i> .—Cantharides, in No. 60 powder, sixty grammes....	60 Gm.
Flexible Collodion, eighty-five grammes.....	85 Gm.
Chloroform, a sufficient quantity,	
To make one hundred grammes..	100 Gm.

Pack the Cantharides firmly in a cylindrical percolator and gradually pour Chloroform upon it until the powder is exhausted. Recover the Chloroform by distillation from a water-bath and evaporate the residue in a tared evaporating dish on a water-bath, until it weighs fifteen grammes. Dissolve this in the Flexible Collodion, and set it aside in a securely corked bottle, and in a cool place, to become clear by settling. Finally, pour off the clear portion from any sediment which may have deposited, and transfer it to bottles, which should be well corked and sealed.

Keep the Cantharidal Collodion in a cool place, remote from lights or fire.

Remarks.—Cantharidal collodion is prepared by percolating cantharides with chloroform, distilling the chloroform from the percolate, and dissolving the remaining extracts in flexible collodion. It is sometimes called *blistering collod*, suggesting its medical use.

COLLODIUM FLEXILE. Flexible Collodion.

<i>Recipe</i> .—Collodion, nine hundred and twenty grammes.....	920 Gm.
Canada Turpentine, fifty grammes.....	50 Gm.
Castor Oil, thirty grammes.....	30 Gm.
To make one thousand grammes..	1000 Gm.

Weigh the ingredients, successively, into a tared bottle, and mix them thoroughly.

Keep the product in cork-stoppered bottles, in a cool place, remote from lights or fire.

Remarks.—The pure pyroxylin layer left on evaporation of ordinary collodion is brittle and apt to crack when placed on the fingers and other joints. Flexible collodion, on the other hand, is made pliable by the addition of Canada turpentine and castor oil.

COLLODIUM STYPTICUM. Styptic Collodion.

<i>Recipe.</i> —Tannic Acid, twenty grammes.....	20 Gm.
Alcohol, five cubic centimeters.....	5 Cc.
Ether, twenty-five cubic centimeters.....	25 Cc.
Collodion, a sufficient quantity,	
To make one hundred cubic centimeters..	100 Cc.

Introduce the Tannic Acid, Alcohol, and Ether into a graduated bottle, agitate the mixture until the Tannic Acid is thoroughly incorporated and partially dissolved, then add enough Collodion to make up the volume to *one hundred cubic centimeters*, and shake occasionally, until the Tannic Acid is completely dissolved.

Keep the product in cork-stoppered bottles, in a cool place, remote from lights or fire.

Remarks.—This represents ordinary collodion made styptic by the addition of 20 per cent. tannic acid. It is useful in stanching the flow of blood from minor wounds.

Note that as collodion is a solution of gun-cotton in a mixture of alcohol and ether, it will be precipitated by the addition of water. Therefore, a prescription containing collodion, tincture of iodine, and water of ammonia cannot be compounded, for as soon as the water of ammonia is added to the collodion, the pyroxylin separates in a wad. This can be remedied by substitution of the spirit of ammonia for water of ammonia.

All the collodions, since they contain ether, must be handled carefully and kept away from the flame.

OLEATES

These are *solutions of metallic oxides or alkaloids in oleic acid*, and, like ointments, produce the physiologic effect of the active ingredient through absorption of the latter on application to the skin by inunction.

They are prepared by dissolving an oxide or an alkaloid in oleic acid, and in making metallic oleates an excess of heat should be avoided, as oleic acid easily reduces the metallic oxides to the metals, particularly when the mixture is heated. The dissolving of the oxides in oleic acid is sometimes a rather difficult matter, and in order to obtain good results the oxide should be freshly precipitated. In making an oleate of an alkaloid, the free alkaloid and not the salts thereof should be used.

All the oleates are, strictly speaking, chemical preparations, since the solvent (oleic acid) forms a salt (oleate) with either a metallic oxide or an alkaloid. Bear in mind, however, that the official oleates, now considered, are not definite chemicals, like sodium oleate (soap), lead oleate (lead plaster), or the other oleates discussed in Chapter XLI. The class we are now considering contain a large amount of oleic acid and, therefore, represent the definite chemical (the true oleate) dissolved in an excess of oleic acid.

SPECIAL NOTES ON OFFICIAL OLEATES**OLEATUM ATROPINÆ. Oleate of Atropine.**

<i>Recipe.</i> —Atropine, two grammes.....	2 Gm.
Alcohol, two cubic centimeters.....	2 Cc.
Oleic Acid, fifty grammes.....	50 Gm.
Olive Oil, a sufficient quantity,	
To make one hundred grammes..	100 Gm.

Triturate the Atropine in a tared mortar with the Alcohol, then add about an equal volume of the Oleic Acid, and, after warming the mortar, stir until the Alcohol has evaporated, add the remainder of the Oleic Acid, and continue stirring until the Atropine is dissolved; then add sufficient Olive Oil to make the product weigh *one hundred grammes*.

Remarks.—This new official is a 2 per cent. solution of atropine.

OLEATUM COCAINÆ. Oleate of Cocaine.

Recipe.—Identical with that for oleate of atropine, except that it contains 5 per cent. of cocaine, and is made with 5 Cc. alcohol to 100 Gm. finished product.

OLEATUM HYDRARGYRI. Oleate of Mercury.

Recipe.—Yellow Mercuric Oxide, in very fine powder,
twenty-five grammes..... 25 Gm.
 Distilled Water, *twenty-five cubic centimeters*..... 25 Cc.
 Oleic Acid, *a sufficient quantity*,
 To make *one hundred grammes*.. 100 Gm.

Triturate the Yellow Mercuric Oxide with the Distilled Water in a tared mortar; add *seventy grammes* of Oleic Acid, and mix thoroughly; warm the mortar to a temperature not exceeding 50° C. (122° F.), stir occasionally until the water has evaporated, then add, if necessary, Oleic Acid to make *one hundred grammes*, and mix thoroughly. Avoid contact with metallic utensils; preserve the Oleate in tightly stoppered bottles.

Remarks.—This is now the only official oleate made from a metallic oxide, oleate of zinc, U. S. P. 1890, having been dropped in the last revision.

OLEATUM QUININÆ. Oleate of Quinine.

Recipe.—Quinine, *twenty-five grammes*..... 25 Gm.
 Oleic Acid, *seventy-five grammes*..... 75 Gm.
 To make *one hundred grammes*.. 100 Gm.

Triturate the Quinine in a warm mortar with a small quantity of the Oleic Acid to a smooth paste. Then add the remainder of the Oleic Acid, previously warmed, and stir frequently, until the Quinine is dissolved.

Remarks.—This 25 per cent. solution of quinine affords an excellent means of administering that important alkaloid to children. Rubbed in at the armpits, it soon gives the physiologic effect of quinine.

OLEATUM VERATRINÆ. Oleate of Veratrine.

Recipe.—Veratrine, *two grammes*..... 2 Gm.
 Oleic Acid, *fifty grammes*..... 50 Gm.
 Olive Oil, *a sufficient quantity*,
 To make *one hundred grammes*.. 100 Gm.

Triturate the Veratrine in a tared mortar with about *five cubic centimeters* of Olive Oil, and, after warming the mortar, add the Oleic Acid, and continue stirring until the Veratrine is dissolved; then add sufficient Olive Oil to make the product weigh *one hundred grammes*.

Remarks.—A 2 per cent. solution of veratrine, which is in little demand.

OLEATUM ZINCI (U. S. P. 1890). Oleate of Zinc.

The former official zinc oleate, a 5 per cent. solution of zinc oxide in oleic acid, is not to be confounded with the commercial powdered zinc oleate, which is discussed on p. 524.

CHAPTER XIV**INFUSIONS, DECOCTIONS, AND TINCTURES****INFUSIONS**

INFUSIONS are *preparations made by the maceration or percolation of vegetable substances with water*. With infusions we begin the study of those preparations made by extraction—by use of the processes of maceration and percolation—rather than by the simple solution of the medicating substance with the diluent.

In some of the official infusions the water used as the extracting solvent is applied cold, but usually it is poured on while boiling hot. It is used cold for such drugs as wild cherry, the active principle of which, hydrocyanic acid, would be driven off or its formation would be prevented by the temperature of boiling water.

Infusions are not much used in this country, American physicians preferring the alcoholic preparations of drugs, such as tinctures and fluidextracts.

They quickly spoil, and should be freshly prepared whenever called for, which is one reason why they have lost their popularity in this country. They are still largely used in England, and the various infusion mugs depicted in works of pharmacy are mostly of English design. Note that while only three infusions are official in the U. S. P., a general formula is given by which all unofficial infusions save those of potent drugs are to be prepared. In U. S. P. 1880 the drug strength of these general infusions was directed to be 10 per cent.; in the revision of 1890 this was reduced one-half, to 5 per cent.; which strength obtains in the present pharmacopœia.

TABLE OF OFFICIAL INFUSIONS

<i>Manufacture.</i>	(ALL GALENIC)	<i>Preparations.</i>
<i>Maceration with hot water.</i>		Infusion of digitalis. Infusion of senna, compound.
<i>Percolation with cold water.</i>		Infusion of wild cherry.

Explanatory of the table given above, it may be said that in making infusions by *hot maceration* the coarsely ground drug is put into a suitable vessel, boiling water is poured upon it, and the maceration permitted to continue a half-hour or an hour.

In England, where a dozen different infusions may be called for in a day's prescription business, those infusions in daily demand are prepared freshly each morning, and the special infusion mugs, such

as are shown in Fig. 164, are in general use. In this country, where a prescription for an infusion comes in but rarely, any suitable glass or porcelain vessel—say, a teapot—can be used, and it is convenient to prepare the infusion by circulatory solution, that is, the ground drug is placed in a small piece of straining cloth and suspended just below the surface of the boiling water. It might be stated that the perforated diaphragm *a* of the infusion mug pictured above is intended for the same purpose. The process of *cold maceration* is sometimes employed in making infusions, but this is done rarely, and is not given official sanction. The process is identical with hot maceration, save that cold water is used. The process of *cold percolation* is directed for making the official infusion of wild cherry, and the manipulation will be found in the official recipe given below. Details of percolation have already been given on pp. 158–162, hence no further remarks are needed here.

The process of *hot percolation* is sometimes employed in making infusions, but is not given official sanction.

It might, however, be mentioned that the first stage in the manufacture of the official fluidextracts of glycyrrhiza (p. 251) and of triticeum (p. 252) is the preparation of an infusion by percolation with hot water.

Infusions are sometimes prepared by *digestion*, that is, by allowing the drug to macerate in water several hours at gentle heat, such as that furnished by a steam radiator or found at the back of a wood-stove or coal-range.

But one word is necessary as to the preparation of infusions by diluting fluidextracts with warm water, a process recommended, for scarcely disinterested reasons, on the labels of the fluidextracts of most manufacturers. That necessary word is *don't!* The only reason an American physician prescribes an infusion is to get only those active principles of a drug which are soluble in water. If he wants those principles which are soluble in alcohol, he prescribes a tincture or a fluidextract. Hence for the pharmacist to dispense an infusion made from a fluidextract is as much a perversion of duty as would be the dispensing of a watery percolate from ginger on a prescription calling for the official tincture.



Fig. 164.—Infusion mug: *a*, Diaphragm.

SPECIAL NOTES ON OFFICIAL INFUSIONS

INFUSA. Infusions.

An ordinary Infusion, the strength of which is not directed by the physician nor specified by the Pharmacopœia, shall be prepared by the following formula:

Recipe.—The Substance, coarsely comminuted, fifty grammes 50 Gm.
 Boiling Water, one thousand cubic centimeters. . . . 1000 Cc.
 Water, a sufficient quantity,
 To make one thousand cubic centimeters . . 1000 Cc.

Introduce the Substance into a suitable vessel provided with a cover, pour upon it the Boiling Water, cover the vessel tightly, and let it stand for half an hour in a warm place. Then strain with expression, and pass enough Water through the strainer to make the Infusion measure *one thousand cubic centimeters*.

Caution.—The strength of Infusions of energetic or powerful substances should be specially prescribed by the physician.

Remarks.—This is the first of the pharmacopœial “general formulas” presented in this book. To be sure, we have already found on p. 716 a general topic, “*aquæ*,” but under that heading was placed only permission to prepare aromatic waters by different processes, but no attempt to give a specific recipe. In the true general formula a recipe is given for the manufacture of every representative of that particular line of preparations, unless a special recipe is given by the pharmacopœia or by the physician, or unless the active ingredient is so potent that the preparation so made is dangerous.

Thus, in the case of the general formula for infusions, a prescription for any infusion, save *digitalis*, wild cherry, and senna compound, or some very powerful drug, is expected to be compounded by the pharmacist according to the general formula. Thus a prescription for 1000 Cc. infusion of *buchu* is to be prepared by macerating 50 Gm. of the bruised drug with 1000 Cc. boiling water, and for any quantity the same proportion of *buchu* to boiling water (5 : 100) is to be preserved.

INFUSUM CINCHONÆ (U. S. P. 1890). Infusion of Cinchona.

Infusion of cinchona, which was dropped in the last revision, is made by percolating 60 Gm. cinchona with a menstruum consisting of 30 Cc. aromatic sulphuric acid and 500 Cc. water, and lastly with enough water to make 1000 Cc.

INFUSUM DIGITALIS. Infusion of Digitalis.

<i>Recipe.</i> — <i>Digitalis</i> , bruised, fifteen grammes.....	15 Gm.
<i>Alcohol</i> , one hundred cubic centimeters.....	100 Cc.
<i>Cinnamon Water</i> , one hundred and fifty cubic centimeters.....	150 Cc.
<i>Boiling Water</i> , five hundred cubic centimeters.....	500 Cc.
<i>Cold Water</i> , a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Upon the *Digitalis*, contained in a suitable vessel, pour the Boiling Water and allow it to macerate for one hour. Then strain, add the Alcohol and Cinnamon Water to the strained liquid, and pass enough Cold Water through the residue on the strainer to make the product measure *one thousand cubic centimeters*. Mix well.

Remarks.—Infusion of *digitalis* contains $1\frac{1}{2}$ per cent. of *digitalis* leaves, the strength of which is exhausted by maceration with boiling water, and the official product flavored with cinnamon water, and enough alcohol added to make the product fairly stable. As noted on p. 716, the manufacture of this infusion from the fluidextract is especially pernicious.

Dose.—8 Cc. (2 fluidrachms).

INFUSUM PRUNI VIRGINIANÆ. Infusion of Wild Cherry.

<i>Recipe.</i> —Wild Cherry, in No. 20 powder, forty grammes....	40 Gm.
Glycerin, fifty cubic centimeters.....	50 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Moisten the powder with *sixty cubic centimeters* of Water, and allow it to macerate for one hour; then pack it firmly in a conical glass percolator, and, having placed the Glycerin in the receiving bottle, gradually pour Water upon the powder and continue percolation until the Infusion measures *one thousand cubic centimeters*. Mix well.

Remarks.—Infusion of wild cherry is made by percolation of wild-cherry bark with cold water, and the finished product represents about 4 per cent. of wild-cherry bark.

Dose.—60 Cc. (2 fluidounces).

INFUSUM SENNÆ COMPOSITUM. Compound Infusion of Senna.

<i>Recipe.</i> —Senna, sixty grammes.....	60 Gm.
Manna, one hundred and twenty grammes.....	120 Gm.
Magnesium Sulphate, one hundred and twenty grammes.....	120 Gm.
Fennel, bruised, twenty grammes.....	20 Gm.
Boiling Water, eight hundred cubic centimeters....	800 Cc.
Cold Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Upon the Senna, Manna, and Fennel, contained in a suitable vessel, pour the Boiling Water, and allow it to macerate for half an hour. Then strain with expression, dissolve the Magnesium Sulphate in the infusion, and again strain. Lastly, add enough Cold Water through the strainer to make the Infusion measure *one thousand cubic centimeters*.

Remarks.—Compound infusion of senna is that old and popular remedy, “senna, manna, and salts,” with a bit of fennel added for flavor. Note its synonym—*black draught*; this must not be confounded with “black drop” (p. 263), as it once was in England, with fatal results.

Dose.—120 Cc. (4 fluidounces).

Doses of Official Infusions

8 Cc. (2 fluidrachms).....	Infusion of digitalis.
60 Cc. (2 fluidounces).....	Infusion of wild cherry.
120 Cc. (4 fluidounces).....	Infusion of senna compound.

The *preservation* of infusions has been the subject of considerable unnecessary writing. By the very character of preparation no attempts should be made to preserve infusions, which should be freshly prepared as required. The addition of any preservative agent to infusions is, of course, out of the question. In England, where the infusions are used very largely, a separate counter is arranged for the use of infusions, and here are placed a line of infusion mugs. At the beginning of each day the contents of these mugs are thrown away and the apparatus carefully washed, and new quantities of the infusions are prepared. No infusion should be dispensed later than twenty-four hours after making.

water, then adding 20 Gm. sassafras bark, 20 Gm. glycyrrhiza, and 10 Gm. mezereum, and letting it macerate two hours. To the strained liquid enough water was added to make 100 Cc.

This decoction, rarely used in this country, is popular in Germany as an alterative.

TINCTURES

Tinctures are *alcoholic solutions of non-volatile substances* obtained by extraction of drugs.

An exception to the statement "alcoholic solutions of non-volatile substances" is found in tincture of iodine, which is made by dissolving the volatile substance iodine in alcohol.

Tinctures prepared from drugs differ from fluidextracts in the matter of strength. All fluidextracts, as we will learn later (p. 239), are adjusted to represent a 100 per cent. product, that is, 100 Cc. represent the active principles of 100 Gm. of a standard drug, while the tinctures are much weaker, none of those official being stronger than 50 per cent. The strength of tinctures varies, ranging from camphorated tincture of opium, which contains but $\frac{4}{10}$ of 1 per cent. opium, its active ingredient, to tincture of lactucarium and other 50 per cent. tinctures. The strength of tinctures underwent a radical change in the last pharmacopœial revision, and the results are much easier to impress upon the memory than were the widely different strengths of the tinctures of the pharmacopœia of 1890.

The following table gives the strength of every tincture recognized by the present pharmacopœia.

STRENGTH OF OFFICIAL TINCTURES

$\frac{4}{10}$ of 1 per cent.: Tincture of opium, camphorated. 0.4 of 1 per cent. opium, 1.6 per cent. total active constituents.

5 per cent.: Tinctures of kino and of musk.

7 per cent.: Tincture of iodine (also 5 per cent. potassium iodide).

10 per cent.: Tinctures of aconite, belladonna leaves, cantharides, capsicum, colchicum seed, digitalis, gelsemium, hyoscyamus, Indian cannabis, lobelia, opium, deodorized opium, physostigma, sanguinaria, squill, stramonium, strophanthus, and veratrum.

20 per cent.: Tinctures of arnica, asafetida, bitter orange, benzoin, calendula, calumba, cardamom, cimicifuga, cinchona, cinnamon, ginger, guaiac, guaiac ammoniated, hydrastis, krameria, myrrh, nutgall, pyrethrum, quassia, quillaja, serpentaria, tolu, valerian, and valerian ammoniated.

50 per cent.: Tinctures of fresh herbs, lactucarium, lemon peel, and sweet orange peel.

Some official tinctures do not adapt themselves to the table just given, so must be given special mention.

<i>Tincture of aloes</i>	represents 10 per cent. aloes and 10 per cent. glycyrrhiza.
<i>Tincture of aloes and myrrh</i>	represents 10 per cent. aloes, 10 per cent. myrrh, and 10 per cent. glycyrrhiza.
<i>Tincture of ferric chloride</i>	represents 35 per cent. solution of ferric chloride or 10 per cent. iron.
<i>Tincture of ipecac and opium</i>	represents 10 per cent. ipecac and 10 per cent. opium.

<i>Tincture of lavender, compound</i>	represents 2½ per cent. oil of lavender and 5 per cent. total active constituents.
<i>Tincture of nux vomica</i>	represents 2 per cent. extract nux vomica, or about 8 per cent. of the drug, or 10 per cent. of the fluidextract.
<i>Tincture of rhubarb</i>	represents 20 per cent. rhubarb and 4 per cent. cardamom.
<i>Tincture of vanilla</i>	represents 10 per cent. vanilla and contains 20 per cent. sugar.

The remaining official tinctures adjust themselves to the following table of strengths:

<i>Name of Tincture.</i>	<i>Percentage of Principal Constituent.</i>	<i>Percentage of Total Active Constituents.</i>
<i>Tincture of benzoïn, compound</i>	10 per cent.	24 per cent.
<i>Tincture of cardamom, compound</i>	2.5 “	6.7 “
<i>Tincture of cinchona, compound</i>	10 “	20 “
<i>Tincture of gambir, compound</i>	5 “	7.5 “
<i>Tincture of gentian, compound</i>	10 “	15 “
<i>Tincture of rhubarb, aromatic</i>	20 “	30 “

Explanatory of the above, let it be said that by “principal constituent” is meant the drug giving the compound tincture its name, and as to “total active constituents” these will be enumerated in the proper place.

Attention should be called to the distinction between “represents” and “contains”; thus it is said above that tincture of vanilla “represents 10 per cent. vanilla and contains 20 per cent. sugar.” This distinction should always be borne in mind in discussing the strength of liquid pharmaceuticals, and it can only be said of any one of these that it “contains” a certain percentage of a drug when that drug is completely dissolved in the liquid, as in the case of the sugar in tincture of vanilla. If the drug is composed of vegetable tissue not entirely soluble in the menstruum, as in the case of the vanilla, then the tincture does not “contain” 10 per cent. vanilla, but merely represents the active principles obtained from 10 per cent. vanilla by extraction. There is another clumsy phase in the glib statement that a certain tincture is 10 per cent.; namely, a true 10 per cent. preparation is one in which all the ingredients are weighed, that is, represents 10 Gm. of drug in 100 Gm. of the finished product. On the other hand, a 10 per cent. tincture contains the activity of 10 Gm. of drug in 100 Cc. of finished tincture. This, however, is a question of splitting hairs, since in making the greater number of the liquid pharmaceuticals of the pharmacopœia the solids are weighed, while the preparation is finished by bringing it to a certain volume.

Summing up the table of strengths of tinctures, we have two 5 per cent. tinctures (three including gambir compound), one 7 per cent. tincture, and four 10 per cent. tinctures. The others are 10 per cent. or 20 per cent., and all those of potent drugs are 10 per cent. except iodine. Before dismissing the question of strength of tinctures, emphasis must be laid on the marked change in the strength of some tinctures in the last revision, through the effort to secure greater uniformity:

<i>One was reduced from 10 per cent. to 5 per cent.</i>	Tincture of kino.
<i>Three were raised from 5 per cent. to 10 per cent.</i>	Tinctures of cantharides, capsicum, and strophanthus.
<i>Nine were reduced from 15 per cent. to 10 per cent.</i>	Tinctures of belladonna leaves, colchicum seed, digitalis, gelsemium, hyoscyamus, Indian cannabis, physostigma, sanguinaria, squill, and stramonium.
<i>One was reduced from 20 per cent. to 10 per cent.</i>	Tincture of lobelia.
<i>One was reduced from 35 per cent. to 10 per cent.</i>	Tincture of aconite.
<i>One was reduced from 40 per cent. to 10 per cent.</i>	Tincture of veratrum.
<i>Seven were raised from 10 per cent. to 20 per cent.</i>	Tinctures of calumba, cardamom, cinnamon, quassia, rhubarb, serpentaria, and tolu.
<i>One was raised from 20 per cent. to 50 per cent.</i>	Tincture of sweet orange peel.

The following tables give lists of official tinctures arranged according to method of manufacture. In passing, it may be said that all of them are galenic preparations.

Official Tinctures Made by Solution or Dilution

<i>Preparation.</i>	<i>Solvent.</i>
Tincture of ferric chloride.....	Alcohol, 650 Cc.; solution of ferric chloride, 350 Cc.
Tincture of iodine.....	Alcohol.
Tincture of ipecac and opium.....	Practically diluted alcohol, U. S. P.
Tincture of nux vomica.....	Alcohol, 750 Cc.; water, 250 Cc.
Tincture of tolu.....	Alcohol.

Official Tinctures Made by Maceration

Menstruum.

Alcohol, U. S. P.....	Tincture of asafetida. Tincture of benzoin. Tincture of benzoin, compound. Tincture of guaiac. Tincture of lemon peel. Tincture of myrrh. Tincture of sweet orange.
Alcohol, 750 Cc.; water, 250 Cc.	Tincture of aloes and myrrh. Tincture of lavender, compound. Tincture of squill.
Diluted alcohol, U. S. P.....	Tincture of aloes. Tincture of arnica. Tincture of gambir, compound. Tincture of musk.
Alcohol, 350 Cc.; water, 650 Cc.....	Tincture of quillaja.
Glycerin, 150 Cc.; alcohol, 650 Cc.; water, 200 Cc.....	Tincture of kino.
Glycerin, 50 Cc.; diluted alcohol, 950 Cc.....	Tincture of cardamom, compound.
Glycerin, 40 Cc.; diluted alcohol, 950 Cc.....	Tincture of opium, camphorated.
Aromatic spirit of ammonia.....	Tincture of guaiac, ammoniated.
Two of the above—arnica and squill—are made by maceration with expression.	

Official Tinctures Made by Percolation

<i>Menstruum.</i>	<i>Preparation.</i>
Alcohol, U. S. P.....	Tincture of calendula. Tincture of cantharides. Tincture of cimicifuga. Tincture of ginger. Tincture of Indian cannabis. Tincture of physostigma. Tincture of pyrethrum. Tincture of veratrum.
Alcohol, 950 Cc.; water, 50 Cc.....	Tincture of capsicum.
Alcohol, 750 Cc.; water, 250 Cc.	Tincture of valerian.
Alcohol, 700 Cc.; water, 300 Cc.	Tincture of aconite.
Alcohol, 650 Cc.; water, 350 Cc.	Tincture of gelsemium. Tincture of hydrastis. Tincture of serpentaria. Tincture of strophanthus. Tincture of vanilla.
Alcohol, 600 Cc.; water, 400 Cc.	Tincture of bitter orange. Tincture of calumba. Tincture of colchicum seed. Tincture of gentian, compound.
Diluted alcohol, U. S. P.....	Tincture of belladonna leaves. Tincture of cardamom. Tincture of digitalis. Tincture of hyoscyamus. Tincture of krameria. Tincture of lobelia. Tincture of opium. Tincture of stramonium.
Alcohol, 350 Cc. ; water, 650 Cc.....	Tincture of quassia.
Alcohol, 200 Cc.; water, 800 Cc.	Tincture of deodorized opium.
Glycerin, 100 Cc.; alcohol, 900 Cc....	Tincture of nutgall.
Glycerin, 75 Cc.; alcohol, 675 Cc.; water, 250 Cc.....	Tincture of cinchona. Tincture of cinchona, compound. Tincture of cinnamon.
Glycerin, 250 Cc.; alcohol, 500 Cc.; water, 200 Cc.....	Tincture of lactucarium.
Glycerin, 100 Cc.; alcohol, 500 Cc.; water, 400 Cc.....	Tincture of rhubarb. Tincture of rhubarb, aromatic.
Acetic acid, 20 Cc.; alcohol, 600 Cc.; water, 400 Cc.....	Tincture of sanguinaria.
Aromatic spirit of ammonia.....	Tincture of valerian, ammoniated.

As to the three methods of preparing tinctures in the table just given, but little need be said at this time. The process of *solution* or *dilution* requires no comment, since full details of manufacture of each tincture so prepared will be given in the special notes.

The processes of *maceration* and of *percolation* have been fully explained in Chapter X.; so here we need only state that a tincture is directed by the pharmacopœia to be made by maceration only when the percolation of the drug is impossible or inconvenient.

This is the case in the tinctures made from resinous drugs, for, should we powder such drugs, pack them in the percolator, and attempt percolation, the mass in the percolator would soon clog and the percolation cease. The percolation of such drugs can be accomplished only if the drug is first mixed with once or twice its weight of clean, dry sand. This manipulation is hardly worth the trouble, and, therefore, the pharmacopœia directs that tinctures of such drugs be

prepared by simple maceration. The details of the pharmacopœial recipe for almost every tincture prepared by maceration are identical, and this is still truer of those tinctures prepared by percolation; hence, in the special notes only the typical recipes will be given in full, and the recipe of other tinctures will be compared with and referred to the typical recipe.

Already on p. 160 we discussed the choice of the appropriate menstrua, so here it need only be said, on comparison of the above table of menstrua with the discussion of the chemical constituents of the drugs employed, found in Part IV., it will be usually found that the drugs requiring strong alcohol for extraction are those grouped among resins, gum-resins, oleoresinous, or volatile oil drugs; that those extracted with glycerinic menstruum are tannin-bearing drugs; that those having acetic acid in the menstruum are drugs containing volatile alkaloids; while the ordinary drug-containing glucosides, neutral principles, or stable alkaloids are extracted with alcohol more or less diluted with water.

There are 64 tinctures recognized by the present pharmacopœia—8 less than were recognized by the pharmacopœia of 1890.

SPECIAL NOTES ON OFFICIAL TINCTURES

TINCTURA ACONITI. Tincture of Aconite.

Recipe.—Aconite, in No. 60 powder (containing not less than 0.5 per cent. of aconitine), *one hundred grammes*.. 100 Gm.
 Alcohol,
 Water, each, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Mix Alcohol and Water in the proportion of *seven hundred cubic centimeters* of Alcohol to *three hundred cubic centimeters* of Water. Moisten the Aconite with *forty cubic centimeters* of this menstruum, transfer it to a percolator, and, without pressing the powder, allow it to stand, well covered, for six hours; then pack it very firmly and pour on enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding menstruum until *one thousand cubic centimeters* are obtained.

Tincture of Aconite, when assayed, should contain in *one hundred cubic centimeters* 0.045 Gm. of aconitine.

Remarks.—The assay of this tincture will be explained in Chapter LV. It should contain $\frac{4.5}{1000}$ of 1 per cent. aconitine.
Dose.—0.6 Cc. (10 minims).

TINCTURA ALOES. Tincture of Aloes.

Recipe.—Purified Aloes, in No. 40 powder, *one hundred grammes*..... 100 Gm.
 Glycyrrhiza, in No. 40 powder, *two hundred grammes*..... 200 Gm.
 Diluted Alcohol, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Macerate the Purified Aloes and Glycyrrhiza in a stoppered container, in a moderately warm place, with *seven hundred and fifty cubic centimeters* of Diluted Alcohol, for seven days, with occasional agitation; then filter through purified cotton or a plain paper filter, and, when the liquid has drained off completely, pass enough Diluted Alcohol through the residue to make *one thousand cubic centimeters* of Tincture.

Remarks.—This represents, besides the chief ingredient, 20 per cent. licorice root. Note that this and all tinctures prepared by maceration are directed to be filtered either through absorbent cotton or through a plain filter, plaited filters not being advised.

Dose.—2 Cc. (30 minims).

TINCTURA ALOES ET MYRRHÆ. Tincture of Aloes and Myrrh.

Recipe.—Made by macerating 100 Gm. purified aloes, 100 Gm. myrrh, and 10 Gm. glycyrrhiza with the menstruum—alcohol, 750 Cc.; water, 250 Cc.—to make 1000 Cc. Details as in making tincture of aloes.

Dose.—2 Cc. (30 minims).

TINCTURA ARNICÆ. Tincture of Arnica.

Recipe.—Arnica, in No. 20 powder, *two hundred grammes*. . . . 200 Gm.
Diluted Alcohol, a sufficient quantity,

To make *one thousand cubic centimeters*. . . 1000 Cc.

Macerate the Arnica with *five hundred cubic centimeters* of Diluted Alcohol in a closed vessel, in a moderately warm place, for three days, with occasional stirring, and express strongly. Repeat this operation twice successively with *two hundred and fifty cubic centimeters* of Diluted Alcohol, macerating for twenty-four hours each time; then, having ascertained the volume of the united expressed liquids, macerate the residual marc for six hours with sufficient menstruum to make approximately *one thousand cubic centimeters*, and express as before. Mix the expressed liquids, filter through paper, and pass sufficient Diluted Alcohol through the filter to make *one thousand cubic centimeters* of Tincture.

Remarks.—This preparation was called *tincture of arnica flowers* by the pharmacopœia of 1890, since at that time the *tincture of arnica root* was made by percolating 100 Gm. arnica root with enough menstruum—alcohol, 650 Cc.; water, 350 Cc.—to make 1000 Cc. In the pharmacopœia of 1890 the tincture of the flowers was made by percolation, but in the last revision its manufacture by maceration and expression was wisely adopted, since the drug is very bulky and highly absorbent; hence percolation means a large waste of menstruum, unless the marc is expressed. The popularity of tincture of arnica as a vulnerary is well known. It is rarely given internally.

Dose.—1 Cc. (15 minims).

TINCTURA ASAFÆTIDÆ. Tincture of Asafetida.

Recipe.—Made by maceration of asafetida for three days with alcohol. Strength, 20 per cent. Details as in making tincture of aloes.

Dose.—1 Cc. (15 minims).

TINCTURA AURANTII AMARI. Tincture of Bitter Orange Peel.

Recipe.—Moisten 200 Gm. of drug in No. 40 powder with 80 Cc. of menstruum—alcohol, 600 Cc.; water, 400 Cc.; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

TINCTURA AURANTII DULCIS. Tincture of Sweet Orange Peel.

Recipe.—Sweet Orange Peel, from the fresh fruit, in thin shavings and cut into narrow shreds, *five hundred grammes*. 500 Gm.
 Alcohol, a sufficient quantity,
 To make *one thousand cubic centimeters*. . 1000 Cc.

Macerate the Sweet Orange Peel, in a stoppered wide-mouthed container and in a moderately warm place, with *one thousand cubic centimeters* of Alcohol, during forty-eight hours, with frequent agitation; then filter through purified cotton, and, when the liquid has drained off completely, gradually pass enough menstruum through the residue to make *one thousand cubic centimeters* of Tincture; finally, filter it through paper.

Remarks.—This tincture is a pleasant flavor, more like a spirit than a tincture.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA BELLADONNÆ FOLIORUM. Tincture of Belladonna Leaves.

Recipe.—Moisten with 100 Gm. of drug in No. 60 powder with 40 Cc. of the menstruum—diluted alcohol; macerate three hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—For the assay of this tincture, see Chapter LV. It should contain $\frac{3}{100}$ of 1 per cent. of mydriatic alkaloids.

Dose.—0.5 Cc. (8 minims).

TINCTURA BENZOINI. Tincture of Benzoin.

Recipe.—Benzoin, in No. 40 powder, *two hundred grammes*. . . 200 Gm.
 Alcohol, a sufficient quantity,
 To make *one thousand cubic centimeters*. . 1000 Cc.

Triturate the Benzoin with *two hundred and fifty cubic centimeters* of Alcohol until a uniform magma is obtained. Transfer this to a stoppered container with the aid of *five hundred cubic centimeters* of Alcohol, and set it aside in a moderately warm place, shaking frequently during three days. Then transfer the mixture to a paper filter, and, when the liquid has drained off completely, pour on enough Alcohol to make *one thousand cubic centimeters* of Tincture.

Remarks.—It will be seen that the above recipe is a slight modification of the maceration process for making tincture of asafetida.

Dose.—1 Cc. (15 minims).

TINCTURA BENZOINI COMPOSITA. Compound Tincture of Benzoin.

Recipe.—Benzoin, in No. 40 powder, *one hundred grammes*. . . 100 Gm.
 Purified Aloes, in No. 40 powder, *twenty grammes*. . . 20 Gm.
 Storax, *eighty grammes*. 80 Gm.
 Balsam of Tolu, *forty grammes*. 40 Gm.
 Alcohol, a sufficient quantity,
 To make *one thousand cubic centimeters*. . 1000 Cc.

Triturate the Benzoin and Purified Aloes with *two hundred and fifty cubic centimeters* of Alcohol until a uniform magma is obtained. Transfer this to a stoppered container with the aid of *five hundred cubic centimeters* of Alcohol, add the Storax and Balsam of Tolu, and set the mixture aside in a moderately warm place, shaking

it frequently during three days; then transfer it to a paper filter, and, when the liquid has drained off completely, pour on enough Alcohol to make *one thousand cubic centimeters* of Tincture.

Remarks.—This useful preparation is similar to, but not identical with, the old-time proprietaries: *friars' balsam*, *Turlington's balsam*, *elixir commendatoris*, or *baume de commandeur*. The latter product contained balsam of Peru, myrrh, and angelica root, in addition to the ingredients of the official tincture. A recipe for the true friars' balsam is found in the National Formulary, but it is usually the custom in this country to dispense compound tincture of benzoin on calls for the preparations whose synonyms have just been given.

Dose.—2 Cc. (30 minims).

TINCTURA CALENDULÆ. Tincture of Calendula.

Recipe.—Moisten 200 Gm. drug in No. 20 powder with 80 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—None is given, since the preparation is used almost entirely externally as a vulnerary.

TINCTURA CALUMBÆ. Tincture of Calumba.

Recipe.—Moisten 200 Gm. drug in No. 20 powder with 100 Cc. of the menstruum—600 Cc. alcohol; 400 Cc. water; macerate twenty-four hours before packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CANNABIS INDICÆ. Tincture of Indian Cannabis.

Recipe.—Moisten 100 Gm. of the drug in No. 40 powder with 50 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in tincture of aconite.

Dose.—0.6 Cc. (10 minims).

TINCTURA CANTHARIDIS. Tincture of Cantharides.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 35 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—0.3 Cc. (5 minims). Used chiefly externally as a constituent of hair-dressings.

TINCTURA CAPSICI. Tincture of Capsicum.

Recipe.—Moisten 100 Gm. of the drug in No. 50 powder with 35 Cc. of the menstruum—950 Cc. alcohol; 50 Cc. water; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—0.5 Cc. (8 minims).

TINCTURA CARDAMOMI. Tincture of Cardamom.

Recipe.—Moisten 200 Gm. of the drug in No. 30 powder with 80 Cc. of the menstruum—diluted alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CARDAMOMI COMPOSITA. Compound Tincture of Cardamom.

Recipe.—Macerate 25 Gm. cardamom; 25 Gm. Saigon cinnamon; 12 Gm. caraway, and 5 Gm. cochineal, ground together to a No. 40 powder, seven days with the menstruum—glycerin, 50 Cc.; diluted alcohol, 950 Cc.; finally using enough diluted alcohol to make 1000 Cc. Details as in making tincture of aloes.

Remarks.—A mnemonic for the active constituents of this compound tincture are the four C's:

Cardamom
Cinnamon
Caraway
Cochineal

This bright-red liquid—the color due to the cochineal—is a pleasant aromatic and carminative.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CIMICIFUGÆ. Tincture of Cimicifuga.

Recipe.—Moisten 200 Gm. of the drug in No. 40 powder with 60 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CINCIONÆ. Tincture of Cinchona.

Recipe.—Cinchona, in No. 60 powder (yielding not less than 4 per cent. of anhydrous ether-soluble alkaloids),
two hundred grammes..... 200 Gm.
 Glycerin, *seventy-five cubic centimeters*..... 75 Cc.
 Alcohol,
 Water, each, a sufficient quantity,
 To make *one thousand cubic centimeters*... 1000 Cc.

Mix the Glycerin with *six hundred and seventy-five cubic centimeters* of Alcohol and *two hundred and fifty cubic centimeters* of Water. Moisten the Cinchona with *eighty cubic centimeters* of the menstruum, transfer it to a percolator, and, without pressing the powder, allow it to stand, well covered, for six hours; then pack it firmly and pour on enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, pouring on first the remainder of the menstruum, and then sufficient of a mixture of Alcohol and Water, made in the same proportions as before, until *one thousand cubic centimeters* of percolate are obtained.

Tincture of Cinchona, when assayed, should contain in *one hundred cubic centimeters* 0.75 Gm. of anhydrous ether-soluble alkaloids of Cinchona.

For Assay, see Chapter LV.

Remarks.—The pharmacopœial recipe is given in full because the percolation in this case differs from that in making tincture of aconite, since the same menstruum is not used through the entire process. That is, in making a liter of tincture, when, after the liter of glycerinic menstruum is used up (for, bear in mind, more than one liter of menstruum has to be used to get a liter of percolate), the percolation is continued with a mixture of alcohol and water (hydro-alcoholic menstruum) until the liter of finished product is attained. Since this statement is applied to all tinctures made by percolation of drug with glycerinic menstruum, the above recipe is typical.

The pharmacopœial assay for this tincture is given in Chapter LV. It should contain $\frac{7.5}{100}$ of 1 per cent. of the ether-soluble cinchona alkaloids.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CINCHONÆ COMPOSITA. Compound Tincture of Cinchona.

Recipe.—Moisten 100 Gm. red cinchona, 80 Gm. bitter orange peel, and 20 Gm. serpentaria, all ground together to a No. 60 powder, with 80 Cc. of the same menstruum used in making tincture of cinchona, and proceed to percolate to 1000 Cc., as in making the latter tincture.

Remarks.—This is sometimes called Huxham's tincture of bark, and is a valuable tonic. It should not be dispensed with iron preparations, since the cinchotannic acid present in the bark reacts with iron to form an inky mixture.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA CINNAMOMI. Tincture of Cinnamon.

Recipe.—Moisten 200 Gm. of the drug in a No. 50 powder with 80 Cc. of the menstruum, the same as used in the cinchona tinctures; macerate six hours before and twenty-four hours after packing, and proceed to make 1000 Cc. tincture by percolation, exactly as in preparing tincture of cinchona.

Dose.—2 Cc. (30 minims).

TINCTURA COLCHICI SEMINIS. Tincture of Colchicum Seed.

Recipe.—Moisten 100 Gm. of the drug in a No. 50 powder with 40 Cc. of the menstruum—alcohol, 600 Cc.; water, 400 Cc.; macerate six hours before and forty-eight hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The assay of this tincture will be found in Chapter LV. It should contain $\frac{4}{100}$ of 1 per cent. of colchicine.

Dose.—2 Cc. (30 minims).

TINCTURA DIGITALIS. Tincture of Digitalis.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 40 Cc. of the menstruum—diluted alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The tincture of digitalis contains an appreciable amount of fat naturally existing in the leaves, and of late years efforts have been made to introduce the so-called fat-free tincture of digitalis. Such a tincture is prepared by first removing the fat from the drug by percolation with chloroform. (See Proceedings of Amer. Phar. Assoc., 1900, p. 530.)

Dose.—1 Cc. (15 minims).

TINCTURA FERRI CHLORIDI. Tincture of Ferric Chloride.

This preparation can best be discussed among the salts of iron. (See p. 557.)

TINCTURA GALLÆ. Tincture of Nutgall.

Recipe.—Nutmeg, in No. 40 powder, two hundred grammes.... 200 Gm.
Glycerin, one hundred cubic centimeters..... 100 Cc.
Alcohol, a sufficient quantity,
To make one thousand cubic centimeters.. 1000 Cc.

Mix the Glycerin with *nine hundred cubic centimeters* of Alcohol. Introduce the Nutgall, without moistening it, into a glass percolator, shaking down the powder evenly and compactly, and pour on sufficient of the menstruum to saturate it and leave a stratum above it. Allow the percolation to proceed slowly, pouring on the remainder of the menstruum, and then sufficient Alcohol to obtain *one thousand cubic centimeters* of Tincture.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA GAMBIR COMPOSITA. Compound Tincture of Gambir.

Recipe.—Macerate 50 Gm. gambir and 25 Gm. Saigon cinnamon both in a No. 50 powder for forty-eight hours, with enough diluted alcohol to make 1000 Cc. tincture. Details as in making tincture of aloes.

Remarks.—This new official replaces the formerly official *tincture of catechu*, being identical with it, save the use of pale catechu (gambir), instead of the catechu from the plant *Acacia catechu*. (See p. 795.)

Dose.—4 Cc. (1 fluidrachm).

TINCTURA GELSEMI. Tincture of Gelsemium.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 35 Cc. of the menstruum—alcohol, 650 Cc.; water, 350 Cc.; macerate six hours before and twenty-four hours after packing; then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—0.5 Cc. (8 minims).

TINCTURA GENTIANÆ COMPOSITA. Compound Tincture of Gentian.

Recipe.—Moisten 100 Gm. gentian; 40 Gm. bitter orange peel, and 10 Gm. cardamom, ground together to a No. 40 powder, with 60 Cc. of the menstruum—alcohol, 600 Cc.; water, 400 Cc.; macerate twelve hours before and twelve hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA GUAIACI. Tincture of Guaiac.

Recipe.—Macerate 200 Gm. of the drug in a No. 40 powder for three days with enough alcohol (U. S. P.) to make 1000 Cc. of the tincture. Details as in making tincture of aloes.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA GUAIACI AMMONIATA. Ammoniated Tincture of Guaiac.

Recipe.—Macerate 200 Gm. guaiac in a No. 40 powder for three days with enough aromatic spirit of ammonia to make 1000 Cc. of finished tincture. Details as in making tincture of aloes.

Remarks.—The pharmacopœial recipes for these two tinctures of guaiac are not identical with that for tincture of aloes, inasmuch as no mention is made of the filtering medium.

Dose.—2 Cc. (30 minims).

TINCTURA HYDRASTIS. Tincture of Hydrastis.

Recipe.—Macerate 200 Gm. of the drug in a No. 60 powder with 60 Cc. of the menstruum—650 Cc. alcohol; 350 Cc. water; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The pharmacopœial assay of this tincture will be given in Chapter LV. It should contain $\frac{1}{10}$ of 1 per cent. of hydrastine.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA HYOSCYAMI. Tincture of Hyoscyamus.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 40 Cc. of the menstruum—diluted alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The pharmacopœial assay of this tincture will be given in Chapter LV. It should contain $\frac{7}{1000}$ of 1 per cent. of mydriatic alkaloids.

Dose.—2 Cc. (30 minims).

TINCTURA IODI. Tincture of Iodine.

This important official will be discussed under Iodine (p. 397).

TINCTURA IPECACUANHÆ ET OPII. Tincture of Ipecac and Opium.

<i>Recipe.</i> —Tincture of Deodorized Opium, one thousand cubic centimeters.....	1000 Cc.
Fluidextract of Ipecac, one hundred cubic centimeters.....	100 Cc.
Diluted Alcohol, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Evaporate the Tincture of Deodorized Opium, in a tared dish on a water-bath, until it weighs *eight hundred grammes*. When it has become cold, add to it the Fluidextract of Ipecac, filter the mixture, and pass enough Diluted Alcohol through the filter to obtain *one thousand cubic centimeters* of Tincture.

Remarks.—This official—sometimes called *tincture of Dover's powder*—is a liquid form of Dover's powder, containing, like it, 10 per cent. ipecac and 10 per cent. opium. It is made from tincture of deodorized opium and fluidextract of ipecac.

Dose.—0.5 Cc. (8 minims).

TINCTURA KINO. Tincture of Kino.

<i>Recipe.</i> —Kino, fifty grammes.....	50 Gm.
Purified Talc, ten grammes.....	10 Gm.
Glycerin, one hundred and fifty cubic centimeters...	150 Cc.
Alcohol, six hundred and fifty cubic centimeters....	650 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Mix the Glycerin with *two hundred cubic centimeters* of Water, and triturate the Kino and Purified Talc with sufficient of the mixture to produce a thin, smooth magma. Transfer this magma to a flask by the aid of the remainder of the mixture, and, having ascertained the weight of the flask and contents, heat it on a water-bath for about one hour; allow the flask and contents to cool, and restore the original weight by the addition of sufficient Water. Then add the Alcohol, mix, and pass the Tincture through a filter of purified cotton, keeping the funnel well covered. Finally, add sufficient Alcohol through the filter to obtain *one thousand cubic centimeters* of Tincture.

Remarks.—The tincture of kino of the former pharmacopœias had to be kept in small well-stoppered and completely filled bottles; else it gelatinizes. It has been found that this gelatinization was due to an enzyme, which can be destroyed by heat, hence the direction in the present recipe to heat the glycerinic magma on a water-bath for an hour. So prepared, the tincture is said to keep indefinitely.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA KRAMERIÆ. Tincture of Krameria.

Recipe.—Moisten 200 Gm. of the drug in a No. 40 powder with 80 Cc. of the menstruum—diluted alcohol; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—This is one of the few tinctures of tannin-bearing drugs made without a glycerinic menstruum.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA LACTUCARII. Tincture of Lactucarium.

<i>Recipe.</i> —Lactucarium, five hundred grammes.....	500 Gm.
Glycerin, two hundred and fifty cubic centimeters...	250 Cc.
Alcohol,	
Purified Petroleum Benzin,	
Diluted Alcohol,	
Water, each, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Beat the Lactucarium in an iron mortar, with clean sand, to a coarse powder, and introduce it into a bottle; add *two thousand cubic centimeters* of Purified Petroleum Benzin, cork the bottle tightly, and set it aside for forty-eight hours, frequently agitating the mixture. Pour the mixture on a double filter and allow it to drain. Wash the residue by gradually adding *fifteen hundred cubic centimeters* of Purified Petroleum Benzin, and allow the Lactucarium to dry by exposing it to a current of air. When it is dry and free from the odor of Benzin, reduce it to powder, using

more sand, if necessary, and pack it moderately in a conical percolator. Mix the Glycerin with *two hundred cubic centimeters* of Water and *five hundred cubic centimeters* of Alcohol, and moisten the powder with *five hundred cubic centimeters* of the mixture. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for twenty-four hours. Then allow the percolation to proceed very slowly, gradually adding, first the remainder of the menstruum, and then Diluted Alcohol, until the Lactucarium is exhausted. Reserve the first *seven hundred and fifty cubic centimeters* of the percolate, evaporate the remainder on a water-bath, at a temperature not exceeding 70° C. (158° F.), to *two hundred and fifty cubic centimeters*, and mix this with the reserved portion. Filter, and add enough Diluted Alcohol through the filter to obtain *one thousand cubic centimeters* of Tincture.

Remarks.—Tincture of lactucarium is interesting in that the lactucarium is first washed with benzin to remove the rubber-like, inert substance it contains. Be sure that the benzin used is of pharmacopœial purity, as most of the commercial benzin is very impure. (See p. 637.)

Note that the drug is not easily exhausted with the menstruum employed, and that in making 1000 Cc., the first 750 Cc. of percolate is reserved and the rest of the percolate evaporated, similar to making fluidextracts.

Dose.—2 Cc. (30 minims).

TINCTURA LAVANDULÆ COMPOSITA. Compound Tincture of Lavender.

<i>Recipe.</i> —Oil of Lavender Flowers, <i>eight cubic centimeters</i> . . .	8 Cc.
Oil of Rosemary, <i>two cubic centimeters</i>	2 Cc.
Saigon Cinnamon, <i>twenty grammes</i>	20 Gm.
Cloves, <i>five grammes</i>	5 Gm.
Myristica, <i>ten grammes</i>	10 Gm.
Red Saunders, <i>ten grammes</i>	10 Gm.
Alcohol,	
Water, each, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters* . . 1000 Cc.

Reduce the Saigon Cinnamon, Cloves, Myristica, and Red Saunders to a No. 50 powder, and macerate this powder for three days in a mixture of *seven hundred and fifty cubic centimeters* of Alcohol and *two hundred and fifty cubic centimeters* of Water, in which liquid the Oils have been dissolved. Then filter, and, when the liquid has drained off completely, pass enough of a mixture of Alcohol and Water, made in the same proportions as before, through the residue on the filter to make *one thousand cubic centimeters* of Tincture.

Remarks.—This tincture is sometimes called “red lavender,” because of its brilliant color, due to the red saunders it contains. It is reputed good in “heart trouble,” although, in truth, its only value is to relieve flatulence.

Dose.—2 Cc. (30 minims).

TINCTURA LIMONIS CORTICIS. Tincture of Lemon Peel.

Recipe.—This is a 50 per cent. alcoholic extract of fresh lemon peel, made exactly as tincture of sweet orange peel.

Remarks.—Tincture of lemon peel is a new official, replacing spirit of lemon of the pharmacopœia of 1890. It is used entirely as a flavor, hence no dose is given.

TINCTURA LOBELIÆ. Tincture of Lobelia.

Recipe.—Moisten 100 Gm. of the drug in a No. 50 powder with 40 Cc. of the menstruum—diluted alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—Expectorant, 1 Cc. (15 minims); emetic, 4 Cc. (1 fluidrachm).

TINCTURA MOSCHI. Tincture of Musk.

<i>Recipe.</i> —Musk, five grammes.....	5 Gm.
Alcohol, forty-five cubic centimeters.....	45 Cc.
Water, forty-five cubic centimeters.....	45 Cc.
Diluted Alcohol, a sufficient quantity,	
To make one hundred cubic centimeters..	100 Cc.

Triturate the Musk with the Water, a little at a time, until a smooth mixture is obtained; transfer this mixture to a bottle and allow it to stand twenty-four hours; add the Alcohol and macerate the mixture for six days, occasionally shaking it. Then filter through a plain paper filter, and, when the liquid has drained off completely, pass enough Diluted Alcohol through the filter to make *one hundred cubic centimeters* of Tincture.

Remarks.—As seen in the recipe given above, this tincture is made by rubbing musk with water, adding alcohol, filtering, and washing residue with diluted alcohol.

It contains 5 per cent. of musk and should be prepared with the utmost care, for when it is required, a fine quality is absolutely essential, it being used chiefly as a stimulant in extreme cases of pneumonia.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA MYRRHÆ. Tincture of Myrrh.

Recipe.—Macerate 200 Gm. of the drug in a moderately coarse powder for three days with enough menstruum—alcohol (U. S. P.)—to make 1000 Cc. Details as in making tincture of aloes.

Remarks.—Myrrh (see p. 857) contains a larger percentage of gummy matter than asafetida, and this gum, which does not dissolve in the strong alcoholic menstruum and which is left on the filter, after making the tincture, has been suggested as a source of label mucilage.

The tincture is used considerably in mouth-washes and is not often given internally.

Dose.—1 Cc. (15 minims).

TINCTURA NUCIS VOMICÆ. Tincture of Nux Vomica.

<i>Recipe.</i> —Extract of Nux Vomica (containing 5 per cent. of strychnine), twenty grammes.....	20 Gm.
Alcohol,	
Water, each, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Extract of Nux Vomica in a sufficient quantity of a mixture of Alcohol and Water, made in the proportion of *seven hundred and fifty cubic centimeters* of Alcohol and *two hundred and fifty cubic centimeters* of water, to make the

solution measure *one thousand cubic centimeters*, and filter through a well-covered filter.

Tincture of Nux Vomica, when assayed, should contain in *one hundred cubic centimeters* 0.1 Gm. of strychnine.

For Assay, see Chapter LV.

Remarks.—This important tincture is made by dissolving the extract in alcoholic menstruum, the finished product containing 2 per cent. of the extract.

In preparing this tincture, great care should be taken to use the U. S. P. powdered extract instead of the soft extract (of the older pharmacopœias) often furnished by manufacturers. This soft extract still contains the fat naturally existing in the nux vomica, while the extract of the present pharmacopœia, being made by percolation of the drug with water and acetic acid, contains no fat. The official extract makes a clear solution with the menstruum directed, while the soft extract will make a milky mixture. Note that the tincture of nux vomica is directed to be assayed for its strength in the alkaloid strychnine (see p. 1036), the amount of strychnine it should contain being $\frac{1}{10}$ of 1 per cent. This follows from the fact that it is to contain 2 per cent. of an extract, which, in turn, must contain 5 per cent. strychnine: 0.02 times 0.05 equals 0.001 or $\frac{1}{10}$ of 1 per cent.

Dose.—0.6 Cc. (10 minims).

TINCTURA OPII. Tincture of Opium.

Recipe.—**Granulated Opium** (containing 12 to 12.5 per cent. of crystallizable morphine), *one hundred grammes*. 100 Gm.
Alcohol,
Water,
Diluted Alcohol, each, *a sufficient quantity,*
 To make *one thousand cubic centimeters*. . 1000 Cc.

Heat *four hundred cubic centimeters* of water to boiling, and pour it on the Granulated Opium contained in a tared vessel, weigh, and stir occasionally during twelve hours; then restore the original weight by the addition of cold Water, add *four hundred cubic centimeters* of Alcohol, pour the mixture into a bottle, and continue the maceration for forty-eight hours, occasionally shaking. Transfer the mixture to a percolator, return the first portion of the percolate until it runs through clear, and, when the liquid ceases to drop, continue the percolation slowly, pouring on sufficient Diluted Alcohol until *one thousand cubic centimeters* are obtained.

Tincture of Opium, when assayed by the process given below, should contain in *one hundred cubic centimeters* not less than 1.2 nor more than 1.25 Gm. of crystallizable morphine.

Remarks.—Note that this tincture—called *laudanum*, *tinctura thebaica*, etc.—is directed to be made from granulated opium, not from the gum, as some druggists prepare it.

The use of granulated opium makes the present official recipe a great improvement over that of the pharmacopœia of 1890, which directed powdered opium. This made percolation very difficult, the finely powdered opium having a tendency to clog the cotton plug, stopping percolation. The morphine assay of this tincture is given in Chapter LV, the pharmacopœia directing that it contain not less than 1.2 per cent. nor more than 1.25 per cent. morphine.

Dose.—0.5 Cc. (8 minims).

TINCTURA OPII CAMPHORATA. Camphorated Tincture of Opium.

<i>Recipe.</i> —Powdered Opium, four grammes.....	4 Gm.
Benzoic Acid, four grammes.....	4 Gm.
Camphor, four grammes.....	4 Gm.
Oil of Anise, four cubic centimeters.....	4 Cc.
Glycerin, forty cubic centimeters.....	40 Cc.
Diluted Alcohol, nine hundred and fifty cubic centimeters.....	950 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Place all the ingredients in a stoppered container and macerate for three days with frequent agitation; then filter the mixture through a well-covered paper filter, adding sufficient Diluted Alcohol through the filter to make *one thousand cubic centimeters* of Tincture.

Remarks.—This popular official is commonly called *paregoric*, the latter word meaning “soothing,” the tincture being originally called “elixir paragoricus,” soothing elixir. Note that it contains $\frac{4}{10}$ of 1 per cent. of opium.

Dose.—8 Cc. (2 fluidrachms).

TINCTURA OPII DEODORATI. Tincture of Deodorized Opium.

<i>Recipe.</i> —Granulated Opium (containing 12 to 12.5 per cent. of crystallizable morphine), one hundred grammes.	100 Gm.
Purified Petroleum Benzin, seventy-five cubic centimeters.....	75 Cc.
Alcohol, two hundred cubic centimeters.....	200 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Heat *five hundred cubic centimeters* of Water to boiling, and pour it on the Granulated Opium contained in a suitable vessel, stirring the mixture frequently during twenty-four hours. Then transfer the mixture to a percolator, return the first portion of the percolate until it runs through clear, and, when the liquid ceases to drop, continue the percolation with Water until the Opium is exhausted. Concentrate the percolate by evaporation on a water-bath until it measures *one hundred and fifty cubic centimeters*, and, when cooled, shake it frequently and vigorously for ten minutes with *sixty-five cubic centimeters* of the Purified Benzin. Separate the Benzin, repeat the shaking out for a few minutes with the remainder of the Benzin, and, having carefully and completely separated this second portion of Benzin, evaporate the remaining liquid in a warm place spontaneously until the odor of Benzin has disappeared, removing the last traces by the heat of a water-bath. Mix the deodorized liquid so obtained with *six hundred cubic centimeters* of Water, filter the mixture through a paper filter, and, having mixed the Alcohol with the filtrate, wash the filter with sufficient Water to make *one thousand cubic centimeters* of Tincture.

Remarks.—This tincture represents an evaporated aqueous percolate of opium, deprived of its odor and of its noxious narcotic principle—narcotine—by shaking with benzin, diluted with water, and then mixed with enough alcohol (200 Cc. to 1000 Cc. of finished tincture) to preserve it. The finished product must represent 10 per cent. of official opium and 1.2 per cent. to 1.25 per cent. morphine. The pharmacopœial assay will be found in Chapter LV.

This product was originally introduced as a proprietary, and because freed from narcotine, was called *denarcotized tincture of opium*. It is supposed to have less unpleasant after-effects than has laudanum.

In passing, attention might be called to the fact that all official

liquid preparations of opium, except paregoric, represent 10 per cent. of the official granulated drug, or 1.2 to 1.25 per cent. morphine.

Dose.—0.5 Cc. (8 minims).

TINCTURA PHYSOSTIGMATIS. Tincture of Physostigma.

Recipe.—Moisten 100 Gm. of the drug in a No. 50 powder with 40 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The pharmacopœial assay of this tincture will be found in Chapter LV. It should contain $\frac{1}{1000}$ of 1 per cent. of the ether-soluble alkaloids of physostigma.

Dose.—1 Cc. (15 minims).

TINCTURA PYRETHRI. Tincture of Pyrethrum.

Recipe.—Moisten 200 Gm. of the drug in a No. 50 powder with 80 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and percolate to 1000 Cc. Details as in making tincture of aconite. Since used almost entirely externally, no dose is given by the pharmacopœia.

TINCTURA QUASSIÆ. Tincture of Quassia.

Recipe.—Moisten 200 Gm. of the drug in a No. 50 powder with 60 Cc. of the menstruum—alcohol, 350 Cc.; water, 650 Cc.; macerate six hours before and twenty-four hours after packing, and percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The recipe is interesting because of the small amount of alcohol used in the menstruum.

Dose.—2 Cc. (30 minims).

TINCTURA QUILLAJÆ. Tincture of Quillaja.

Recipe.—Quillaja, in No. 20 powder, two hundred grammes . . . 200 Gm.
 Alcohol, three hundred and fifty cubic centimeters . . . 350 Cc.
 Water, a sufficient quantity,
 To make one thousand cubic centimeters . . 1000 Cc.

Boil the Quillaja in a covered vessel with *eight hundred cubic centimeters* of Water for fifteen minutes, strain while hot, and wash the residue on the strainer with *two hundred cubic centimeters* of Water, previously heated to boiling. Then evaporate the strained liquid to *six hundred cubic centimeters*, allow it to cool, add the Alcohol, and set it aside for twelve hours. Decant the clear liquid, filter it through paper, then pour the residue on the filter, and, when the liquid ceases to drop, wash the filter with sufficient Water to make *one thousand cubic centimeters* of Tincture.

Remarks.—This is an aqueous decoction of soap bark with enough alcohol (350 Cc. to the 1000) added to preserve it. The preparation is used as an emulsifying agent (see Emulsions, Chapter LV.), but it should never be used for that purpose except under direct orders of a physician, since the saponin of the bark is distinctly poisonous. It is used as a “gum foam” at the soda fountain, and this use is less objectionable than as an emulsifying agent, since the amount administered with each glass of soda-water is infinitesimal. The best gum

foam is made by percolating 200 Gm. powdered soap bark with a menstruum composed of 750 Cc. water and 250 Cc. glycerin until 1000 Cc. of percolate are obtained.

TINCTURA RHEI. Tincture of Rhubarb.

Recipe.—Moisten 200 Gm. rhubarb and 40 Gm. cardamom, ground together to a No. 40 powder, with 90 Cc. of the menstruum—alcohol, 500 Cc.; water, 400 Cc.; glycerin, 100 Cc.; macerate twelve hours before and twenty-four hours after packing, and then percolate to 1000 Cc., using as menstruum at the end, alcohol, 500 Cc.; water, 400 Cc. Details as in making tincture of cinchona.

Remarks.—Note that this tincture contains cardamom besides rhubarb; one of the few cases where a preparation containing more than one drug is not pharmacopœially termed “compound.”

Dose.—4 Cc. (1 fluidrachm).

TINCTURA RHEI AROMATICA. Aromatic Tincture of Rhubarb.

Recipe.—Moisten 200 Gm. rhubarb, 40 Gm. Saigon cinnamon, 40 Gm. cloves, and 20 Gm. nutmeg, ground together to a No. 40 powder, with 90 Cc. of the menstruum—alcohol, 500 Cc.; water, 400 Cc.; glycerin, 100 Cc.; macerate twelve hours before and twenty-four hours after packing, and then percolate to 1000 Cc., using as menstruum at the end, alcohol, 500 Cc.; water, 400 Cc. Details as in making tincture of cinchona.

Remarks.—Note the aromatic constituents of this tincture, which is rarely prescribed, and which is official chiefly because used in making the official aromatic syrup of rhubarb (p. 198).

Dose.—2 Cc. (30 minims).

TINCTURA RHEI DULCIS. Sweet Tincture of Rhubarb.

This former official is made by percolating a mixture of rhubarb, 100 Gm., glycyrrhiza, 40 Gm., anise, 40 Gm., and cardamom, 10 Gm., with the menstruum—glycerin, 100 Cc.; alcohol, 500 Cc.; water, 400 Cc.—until 1000 Cc. of tincture are obtained. Details as in making tincture of rhubarb.

TINCTURA SANGUINARIÆ. Tincture of Sanguinaria.

Recipe.—Sanguinaria, in No. 60 powder, one hundred grammes..... 100 Gm.
 Acetic Acid, twenty cubic centimeters..... 20 Cc.
 Alcohol,
 Water, each, a sufficient quantity,
 To make one thousand cubic centimeters.. 1000 Cc.

Mix six hundred cubic centimeters of Alcohol with four hundred cubic centimeters of Water. Moisten the Sanguinaria with the Acetic Acid and thirty cubic centimeters of this menstruum, transfer it to a percolator, and, without pressing the powder, allow it to stand, well covered, for six hours; then pack it firmly and pour on enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for twenty-four hours. Then allow the percolation to proceed slowly, pouring on sufficient menstruum to obtain one thousand cubic centimeters of Tincture.

Remarks.—This is the only official tincture extracted with menstruum containing acetic acid, and it is worth noting that 1000 Cc. of finished tincture contains but 20 Cc. of the acid, and that all this is mixed with the menstruum used in moistening the drug; that all the menstruum used in percolation is hydro-alcoholic.

Dose.—1 Cc. (15 minims).

TINCTURA SCILLÆ. Tincture of Squill.

Recipe.—Squill, in No. 20 powder, one hundred grammes.... 100 Gm.

Alcohol,

Water, each, a sufficient quantity,

To make one thousand cubic centimeters. . 1000 Cc.

Mix seven hundred and fifty cubic centimeters of Alcohol with two hundred and fifty cubic centimeters of Water. Macerate the Squill with six hundred cubic centimeters of the menstruum, in a closed vessel, in a moderately warm place for three days, occasionally stirring, and express strongly. Repeat this operation with three hundred cubic centimeters of menstruum, macerating one day before expression; and, finally, macerate the residue for six hours in sufficient menstruum to make the united expressed liquids measure about one thousand cubic centimeters. Express as before, mix the expressed liquids, filter through paper, and pass sufficient menstruum through the filter to make one thousand cubic centimeters of Tincture.

Remarks.—It will be seen from the above recipe that this tincture is made by maceration and expression similar to the recipe for tincture of arnica. Since the two processes differ in some details, the pharmacopœial recipe for each is reproduced in full.

Dose.—1 Cc. (15 minims).

TINCTURA SERPENTARIÆ. Tincture of *Serpentaria*.

Recipe.—Moisten 200 Gm. of the drug in a No. 50 powder with 60 Cc. of the menstruum—alcohol, 650 Cc.; water, 350 Cc.; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA STRAMONII. Tincture of *Stramonium*.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 40 Cc. of the menstruum—diluted alcohol (U. S. P.); macerate three hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—The pharmacopœial assay of this tincture will be found in Chapter LV. It should contain $\frac{3}{100}$ of 1 per cent. of mydriatic alkaloids.

Dose.—0.5 Cc. (8 minims).

TINCTURA STROPHANTHI. Tincture of *Strophanthus*.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 50 Cc. of the menstruum—alcohol, 650 Cc.; water, 350 Cc.; macerate six hours before and forty-eight hours after packing, and then percolate to 1000 Cc. Details as in tincture of aconite.

Remarks.—The strength of this tincture was increased in the last revision from 5 to 10 per cent.

Dose.—0.5 Cc. (8 minims).

TINCTURA TOLUTANA. Tincture of Tolu.

Recipe.—Balsam of Tolu, two hundred grammes..... 200 Gm.

Alcohol, a sufficient quantity,

To make one thousand cubic centimeters.. 1000 Cc.

Macerate the Balsam of Tolu in *nine hundred cubic centimeters* of Alcohol, shaking frequently until dissolved; then filter through paper, and wash the filter with sufficient Alcohol to make *one thousand cubic centimeters* of Tincture.

Remarks.—Unlike benzoin and other resinous drugs from which tinctures are prepared, tolu is practically completely soluble in alcohol; hence we can consider the above process as one of solution and not of maceration. The tincture is used in making the official *Syrup of Tolu* (p. 202).

Dose.—2 Cc. (30 minims).

TINCTURA VALERIANÆ. Tincture of Valerian.

Recipe.—Moisten 200 Gm. of the drug in a No. 60 powder with 60 Cc. of the menstruum—alcohol, 750 Cc.; water, 250 Cc.; macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Dose.—4 Cc. (1 fluidrachm).

TINCTURA VALERIANÆ AMMONIATA. Ammoniated Tincture of Valerian.

Recipe.—Identical with that for tincture of valerian, save in the menstruum, aromatic spirit of ammonia being employed.

Remarks.—Note that this is one of the two official tinctures made with aromatic spirit of ammonia as the menstruum, the other being ammoniated tincture of guaiac.

Dose.—2 Cc. (30 minims).

TINCTURA VANILLÆ. Tincture of Vanilla.

Recipe.—Vanilla, cut into small pieces and bruised, *one*

hundred grammes..... 100 Gm.

Sugar, in coarse powder, two hundred grammes.... 200 Gm.

Alcohol,

Water, each, a sufficient quantity,

To make one thousand cubic centimeters.. 1000 Cc.

Mix *six hundred and fifty cubic centimeters* of Alcohol with *three hundred and fifty cubic centimeters* of Water. Macerate the Vanilla in *five hundred cubic centimeters* of the mixture for twelve hours; then drain off the liquid and set it aside. Transfer the Vanilla to a mortar, beat it with the Sugar into a uniform powder, then pack it in a percolator and pour upon it the reserved liquid. When this has disappeared from the surface, continue the percolation by gradually pouring on sufficient menstruum to make *one thousand cubic centimeters* of Tincture.

Remarks.—This—the only official tincture containing sugar—is made by beating up macerated vanilla-beans with sugar, packing,

and then percolating. This tincture of vanilla represents a fine grade of the flavoring *essence of vanilla*. What is commonly sold under this name, however, is a concoction of various inferior flavors, such as coumarin and possibly some vanillin. Another point in connection with the manufacture of the tincture of vanilla is that, in order to secure a really fine tincture of vanilla, it is essential to use a good quality of vanilla-bean. The inferior Tahiti beans can never yield anything but a poor product.

TINCTURA VERATRI. Tincture of Veratrum.

Recipe.—Moisten 100 Gm. of the drug in a No. 60 powder with 40 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—This tincture differs radically from the tincture of veratrum viride of the pharmacopœia of 1890. The present official tincture can be made from either white or green hellebore, and is a 10 per cent. preparation, while the tincture of 1890 was 40 per cent. and made from green hellebore only. The latter closely simulated *Norwood's tincture of veratrum*, formerly largely prescribed.

Dose.—1 Cc. (15 minims).

TINCTURA ZINGIBERIS. Tincture of Ginger.

Recipe.—Moisten 200 Gm. of the drug in a No. 50 powder with 60 Cc. of the menstruum—alcohol (U. S. P.); macerate six hours before and twenty-four hours after packing, and then percolate to 1000 Cc. Details as in making tincture of aconite.

Remarks.—This is the well-known *essence of ginger*. It represents 20 per cent. of the drug, and is made with strong alcohol. When mixed with water, it forms a milky mixture, due to the separation of the resin. The so-called water-soluble ginger is the tincture rubbed with magnesium carbonate and enough water to precipitate much of the resin.

Dose.—2 Cc. (30 minims).

TINCTURÆ HERBARUM RECENTIUM. Tinctures of Fresh Herbs.

Tinctures of Fresh Herbs, when not otherwise directed, are to be prepared according to the following formula:

Recipe.—The Fresh Herb, cut, bruised, or crushed, *five*
hundred grammes..... 500 Gm.
 Alcohol, *one thousand cubic centimeters*..... 1000 Cc.

Macerate the Herb with the Alcohol in a closed vessel in a moderately warm place during fourteen days, with occasional stirring; then strongly express the liquid and filter it through paper.

Tinctures of fresh herbs are a line of preparations made from freshly gathered plants, resembling the *juices* (Succi) of the British Pharmacopœia. They represent 50 per cent. of the fresh herb, 1000 Cc. being made from 500 Gm. of herb. They are rarely used in this country.

The Doses of Official Tinctures

0.1 Cc. ($\frac{1}{2}$ minim).....	Tincture of iodine.
0.3 Cc. (5 minims).....	Tincture of cantharides.
0.5 Cc. (8 minims).....	Tinctures of belladonna leaves, capsicum, ferric chloride, gelsemium, ipecac and opium, opium, deodorized opium, stramonium, and strophanthus.
0.6 Cc. (10 minims).....	Tinctures of aconite, Indian cannabis, and nux vomica.
1.0 Cc. (15 minims).....	Tinctures of arnica, asafetida, benzoin, digitalis, lobelia, myrrh, physostigma, sanguinaria, squill, and veratrum.
2.0 Cc. (30 minims).....	Tinctures of aloes, aloes and myrrh, benzoin compound, cinnamon, colchicum seed, ginger, guaiac ammoniated, hyoscyamus, lactucarium, lavender compound, quassia, rhubarb aromatic, tolu, and valerian ammoniated.
4.0 Cc. (1 fluidrachm).....	Tinctures of bitter orange, calumba, cardamom compound, cimicifuga, cinchona, cinchona compound, gambir compound, gentian compound, guaiac, hydrastis, kino, krameria, musk, nutgall, rhubarb, serpentaria, sweet orange, and valerian.
8.0 Cc. (2 fluidrachms).....	Tincture of opium, camphorated.

CHAPTER XV

FLUIDEXTRACTS, WINES, OLEORESINS, AND VINEGARS

FLUIDEXTRACTS

FLUIDEXTRACTS are alcoholic liquid preparations made by percolation, representing in each cubic centimeter the activity of one gramme of drug; in other words, they are 100 per cent. tinctures.

The fluidextracts of the present pharmacopœia, of the strength of one gramme to the cubic centimeter, are 5 per cent. weaker than those directed by the pharmacopœia of 1870, which represented a troy ounce of drug to the fluidounce. The difference in these strengths is apparent when we appreciate the fact that while the gramme of water is always of the same weight as the cubic centimeter, the troy ounce of water will weigh 480 grains, against the 455.7 grains, which is the weight of the fluidounce of water.

Fluidextracts can be considered in the light of concentrated tinctures, and many official drugs have both the tincture and the fluidextract recognized by the pharmacopœia. The advantage which the fluidextract possesses over tinctures and other preparations are: 1. That they possess a definite strength of 100 per cent., the same as that of the product from which derived. In that way the physician, knowing the dose of the drug, has no difficulty in knowing the dose of the fluidextracts. 2. Fluidextracts are concentrated, a very small quantity of fluidextract sufficing, where a much larger

quantity, sometimes ten times the amount; of a tincture will be needed. 3. Fluidextracts are permanent; being made with alcoholic menstrua, they keep perfectly, and, in fact, frequently becoming better as they age.

HISTORY

The fluidextracts are essentially American products, the first fluidextracts known being recognized by the United States Pharmacopœia of 1850. Seven so-called fluidextracts were mentioned in that standard, and of the seven, but one was a true fluidextract. Of the others, four were concentrated syrups and two were oleoresins. In the pharmacopœia of 1860 twenty-five fluidextracts were recognized, and of these, fifteen were fluidextracts in the modern sense of the term.

Manufacture of Fluidextracts.—Four general processes have been suggested for the manufacture of fluidextracts.

Percolation, with Partial Evaporation.—The drug is moistened with the required quantity of menstruum and macerated in the percolator for a certain length of time, and then percolated with the menstruum until exhausted.

It is practically impossible, by the ordinary process of percolation, to exhaust 100 Gm. of drug with the first 100 Cc. of percolate; in most cases 100 Gm. are not exhausted until about 500 Cc. of percolate have been obtained. Accordingly, in making fluidextracts we percolate the drug until it is exhausted, and instead of evaporating the entire percolate down to 100 Cc., we reserve the first 80 Cc. (approximately) of percolate, subjecting to the heat of evaporation only the balance of percolate.

The first portion of the percolate, usually representing in cubic centimeters 80 per cent. of the weight of the drug in grammes (example: 80 Cc. of percolate from 100 Gm. of drug; 800 Cc. of percolate from 1000 Gm. of drug, etc.), is put to one side and labeled "reserved portion." The rest of the percolate, called "weak percolate," is then placed in an appropriate distilling apparatus in order to recover the alcohol, and when the alcohol is distilled, the residue is evaporated to a soft extract on a water-bath. This soft extract is then dissolved in the reserved portion, and enough menstruum is added to bring the fluid-extract up to the number of cubic centimeters equal to the number of grammes of the drug of which we started. Of the details of this process but two salient points need be mentioned. It will be noticed that we spare the first 80 per cent. of percolate from the heat, to which we subject the remainder of the percolate. This is because it is estimated that this 80 per cent. of percolate contains the active principles of at least 80 per cent. of the drug, and since it is without question that even the heat of the water-bath may possibly modify some of the delicate constituents of the drug, it has been deemed advisable to expose the smallest possible quantity of the soluble part of the drug to such temperature. A second point is that we evaporate the weak percolate to a soft extract instead of merely to a point where it, plus the reserve portion, would measure the number of cubic centimeters of fluid equal to the number of grammes of drug with which we started out. The reason for this is because the liquid part

of such a residue consists wholly of water, and that there would be danger that adding the liquid to the reserve portion would cause a precipitation of some of the constituents of the reserve portion.

This process is not a satisfactory one either for the manufacturer or for the retailer. For the retailer, it is a tedious operation, requiring a large amount of time for evaporation of the reserve portion, and especially when the menstruum is a diluted alcohol. The second disadvantage of the process is the use of heat, which tends to dissociate some of the plant principles.

The above process is essentially the one for the manufacture of official fluidextracts given in the body of the present pharmacopœia, although the pharmacopœial recipes differ from this process in some details.

For instance, there are only seven of the official fluidextracts the recipe of which directs distillation of the alcohol from the weak percolate. Of these, under five fluidextracts (*calumba*, *grindelia*, *mezereum*, *podophyllum*, and *rubus*) the directions are given to distil on water-bath, but no mention is made as to the temperature at which the residue, left after distillation, is to be evaporated; while of the other two (*Indian cannabis* and *nux vomica*) we are told to "distil off the alcohol from the remainder by means of a water-bath and evaporate the residue at a temperature not exceeding 50° C." In other words, in but seven cases is distillation directed. Of the others, the weak percolate of one fluidextract (*rhubarb*) is directed to be evaporated at a temperature not exceeding 70° C.; 28 are to be evaporated at 50° or under, while with 46 no mention is made of the maximum temperature for evaporation. Not a word, however, as to distillation of alcohol.

Taking up this point, it might be said that in the 46 cases where no temperature for evaporation is given, permission to distil the alcohol may be implied, but if this is the case, why is distillation specified in seven cases and omitted in the others? If the silence as to distilling the alcohol is to be considered a prohibition, then the official process is worthless, since no one could profitably prepare a fluidextract if the large amount of alcohol contained in the weak percolate were allowed to escape. As to the 28 fluidextracts, the weak percolates of which are to be evaporated at a temperature of 50°, it must be said that this process is impossible for the retailer. Assuming the intention is to recover the alcohol by distillation, that operation can take place at 50° C. only when carried on in a vacuum pan (p. 92), a piece of apparatus the cost of which precludes its use anywhere save in large factories. If the alcohol is to be evaporated and not distilled, the same question as to costly waste raised above applies here; while the simple act of evaporation of the weak percolate in open air at 50° C. is a task so tedious as to be almost impossible.

Fortunately for the retail pharmacist, there is another process for making fluidextracts permitted by the pharmacopœia—a process that is entirely feasible—the process of repercolation.

2. Percolation with Incomplete Exhaustion.—This process was proposed in order to avoid the disadvantages of the process just described, by doing away with the weak percolate. In this process 1000 Gm. of the drug are taken, moistened, packed, and carefully

percolated until 750 Cc. of percolation are obtained. The marc is then thrown away and the operation is discontinued.

The explanation of this process is that the first 750 Cc. of percolate from the 1000 Gm. of the drug is apt to contain the active principles of at least 75 per cent. of the drug in question. If this be true, the percolate would be a 100 per cent. preparation, and on this assumption the plan has been carried out. The weakness of this process is seen in the fact that there is no proof of the amount of extractive matter contained in the percolate. If the drug is very carefully packed and the percolation proceeds very slowly, the 750 Cc. of percolate may contain more than the active principles of the 750 Gm. of the drug. On the other hand, if the percolation is carried on carelessly, a much weaker product will result.

3. *Repercolation*.—This process was devised by Dr. E. R. Squibb as a means of doing away with the use of heat, which is the disadvantage of the official process, and yet securing a preparation of strength more definite than is obtained by the process of percolation with incomplete exhaustion.

The process which is given official sanction in the introduction to the pharmacopœia is of such importance that a detailed explanation is here given.

Suppose we want to make 1000 Cc. fluidextract of gentian: we take 1000 Gm. drug, and the first thing we do with it is to divide it into four portions of 250 Gm. each. The first 250 Gm. we moisten with the pharmacopœial menstruum, pack, macerate, and percolate, just as in the regular official process, with the sole exception that the percolate, as it drops, is collected into several portions; the first 200 Cc.—dark, heavy, and concentrated—being set aside as finished fluidextract, and the next portions of 250 Cc. each put into bottles and labeled respectively “weak percolate No. 1,” “weak percolate No. 2,” “weak percolate No. 3,” and so on until exhaustion. The writer’s personal experience showed that, on an average, 250 Gm. drug is not completely exhausted until 1250 to 1500 Cc. of percolate have dropped, and it was his rule to use a 500-Cc. bottle for weak percolate No. 4, and to let the last percolate drop therein until the drug was completely exhausted.

As the result of our first percolation we have, from 250 Gm. gentian:

Fluidextract.....	200 Cc.
Weak percolate No. 1.....	250 Cc.
Weak percolate No. 2.....	250 Cc.
Weak percolate No. 3.....	250 Cc.
Weak percolate No. 4 (say).....	350 Cc.

Why do we call the first 200 Cc. finished fluidextract? Simply because experience has shown that, with ordinary care, the first 200 Cc. of a percolate from 250 Gm. drug contains the active principles of at least 200 Gm. of that drug. If the first 200 Cc. of percolate contain the active principles of 200 Gm. drug, the active principles of the remaining 50 Gm. drug will be found in the 1100 Cc. of weak percolates No. 1, 2, 3, and 4 and chiefly in percolates No. 1 and 2. Now we are ready to begin with the second batch of 250 Gm. of ground gentian. We moisten it, not with official menstruum, but with weak percolate

FLUIDEXTRACTS, WINES, OLEORESINS, AND VINEGARS

No. 1 of the previous operation. Then we pack and macerate, then pour in weak percolate No. 2, and after it has soaked in, we pour on weak percolate No. 3, and as that too sinks down as the liquid drops below, we pour on weak percolate No. 4. When that has gone through, we begin to employ the official menstruum. In the meanwhile the liquid dropping from the percolator has been collected in several portions. The first 250 Cc. has been set aside as finished fluidextract. Note that this time we do not set aside the first 200 Cc. as finished fluidextract, as we did in the previous case, but 250 Cc.; for we began percolation this time, not with official menstruum, but with those percolates from the first batch of drug, containing the active principles of about 50 Gm. of said drug. Hence the first 250 Cc. of percolate from the second batch of gentian are almost sure of containing the active principles of 250 Gm. of drug, and are, therefore, set aside as finished fluidextract. #

By this time the bottles that had contained weak percolates No. 1 and 2 are empty, and as the next 250 Cc. percolate drops, it is poured into bottle No. 1; the next batch into bottle No. 2, and so on until exhaustion, when we will have in the four original bottles weak percolates No. 1, 2, 3, and 4.

The percolation of batches No. 3 and 4, 250 Gm. each, of gentian, will be a repetition of the handling of batch No. 2. In both cases the first 250 Cc. of percolate are set aside as finished fluidextract, and, when batch No. 4 is exhausted, the weak percolates No. 1, 2, 3, and 4, properly labeled, are placed in some dark, cool place, there to remain until the next time that the fluidextract must be made. The quantity given in above illustration is purely proportional and, of course, does mean that 950 Cc. fluidextract must be made each time. The 950 Cc. fluidextract—that is, the four portions 200 Cc. plus 250 Cc. plus 250 Cc. plus 250 Cc.—is to be carefully mixed and placed in proper container, and, of course, the next time the fluidextract is to be made from the weak percolates, the amount of the finished product will be 1000 Cc. not

In conclusion let it be said the best way to get the absolutely correct view of repercolation is by a personal performance of the operation. When tried, it will be found no more difficult than percolating a tincture, and, as a matter of fact, every retail pharmacist should make at least a dozen of his most used fluidextracts, and by this method.

If the time the average druggist wastes in entertaining the drug-store loafer were used in this manner, the druggist would have a larger bank account at the end of the year.

This fact cannot be too strongly urged. Do not believe the slick-tongued drummer who tells you that his firm can make fluidextracts better than you can, for he is simply romancing. And from the financial side, personal experience has shown the writer that fluidextracts can be made by the retailer from 25 to 50 per cent. less than the cost of the same product of a reliable house—and, of course, only those of the most reliable firms should be taken.

The greatest objection to repercolation is the fact that every 1000 Cc. of fluidextract made means the stowing away of about 1100 Cc. of weak percolate, which can be used for nothing else; meaning

usually at least 500 Cc. alcohol—capital from which no interest accrues. On the other hand, the process affords an exceedingly easy way of making small quantities—say, 250 Cc.—of the fluidextracts, usually bought by the pound, and thus the excess in stock of weak percolates can be counterbalanced by the diminution in the stock of the fluid-extracts themselves.

4. *Percolation and Maceration with Hydraulic Pressure.*—This process was introduced by a firm of manufacturing pharmacists, but has been abandoned by them in favor of repercolation, which is employed by almost all large manufacturers.

The process consists of taking a large quantity of the drug—say, 1000 kilos,—macerating with a lesser quantity of menstruum,—say, 300 liters,—and, after a certain time, pressing this liquid by hydraulic pressure. The press cake is returned to the macerating vat, moistened with another 300 liters of liquid, which is pressed out. The process is then repeated with a sufficient quantity of menstruum, so that when it is pressed the third time the quantity of pressed liquid, or official fluidextract, will amount to 1000 liters.

Besides the four general processes just described, there are several methods whereby certain special fluidextracts are prepared. Thus, the fluidextracts from some drugs which are easily exhausted can be prepared by *simple percolation*. Thus, the present official recipes for fluidextract of wild cherry and for fluidextract of squill direct simple but *careful* percolation of a kilogramme of drug until one liter of percolate is obtained.

Two other official extracts are exhausted by *hot infusion*, the aqueous percolates being evaporated to the required volume and preserved with alcohol. Due attention will be given below to these special processes.

Preservation of Fluidextracts.—Fluidextracts keep well, as a rule, and the precipitate, often occurring, is almost invariably inert. Do not think that home-made fluidextracts are poor because there is a sediment in them after a few months. The manufacturers' fluid-extracts do the same thing, only they are kept in the factory long enough to allow complete precipitation; then they are filtered and bottled.

It must be borne in mind, however, that in some cases the precipitate formed in fluidextracts contains a valuable constituent, and in this case the precipitate should be retained. Unfortunately, we have as yet no table showing us which precipitates should be retained and which rejected, and, therefore, at present it is customary always to dispense fluidextracts in a clear form.

SPECIAL NOTES ON THE OFFICIAL FLUIDEXTRACTS

There is such great sameness in most of the 85 official fluidextracts that what is said about one of them applies with equal force to almost all the others. Their strength is identical; their manufacture, as to minor details, is similar, with only a few exceptions to break the monotony. Their medical properties are those of the drugs from which they are derived, and will be left for discussion—as in all other galenics—until the constituent drug is considered. Their

doses, which will be grouped at the close of this section, are usually the same as the constituent drug, the only notable exception being the case of fluidextract of senna (which see).

Hence, while there are more fluidextracts than any other class of preparations, they can be discussed in a comparatively small space, and instead of giving, as with the other galenics, a table of preparations followed by specific mention of each fluidextract, we can discuss the few typical fluidextracts, placing the others in tabular form under the appropriate type.

Before taking up the individual fluidextract, attention must be called to the change in nomenclature adopted at the last revision. The pharmacopœia of 1890, and those prior to that date, gave as English name the two words "extractum fluidum" (examples, fluidextract of aconite—*Extractum Aconiti Fluidum*). The new pharmacopœia gives as English and Latin names respectively, fluidextract and fluidextractum (example, Fluidextract of Aconite and Fluidextractum Aconiti).

The only reason given for this change was in order to avoid mixing the extracts and fluidextracts in the body of the pharmacopœia. Now representatives of the class extractum come in regular order many pages removed from the representatives of the other class, fluidextracta.

FLUIDEXTRACTS MADE BY PERCOLATION AND PARTIAL EVAPORATION

Note.—The pharmacopœial recipe for fluidextracts made this way is inserted only in deference to our standard, as mentioned on p. 241. The only way for the retail pharmacist to prepare these is by repercolation, permission to do this being given by the pharmacopœia. (Introduction to U. S. P. VIII, p. lii.)

I. FLUIDEXTRACTS WITH HYDRO-ALCOHOLIC MENSTRUUM

FLUIDEXTRACTUM ACONITI. Fluidextract of Aconite.

Recipe.—Aconite, in No. 60 powder, *one thousand grammes*. . 1000 Gm.

Alcohol,

Water, each, *a sufficient quantity*,

To make about *one thousand centimeters*. . 1000 Cc.

Mix *seven hundred and fifty cubic centimeters* of Alcohol with *two hundred and fifty cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding menstruum, using the same proportions of Alcohol and Water as before, until the Aconite is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder, in a porcelain dish, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, mixing thoroughly.

Assay *ten cubic centimeters* of this liquid; from the results thus obtained, ascertain by calculation the amount of aconitine in the remainder of the liquid, add to this enough menstruum to make each *one hundred cubic centimeters* of the finished Fluidextract contain 0.4 Gm. of aconitine.

For Assay, see Chapter LV.

Remarks.—This fluidextract is taken as the type simply because it is the first representative of the class appearing in the pharmacopœia. In studying the recipe given above it will be found that the menstruum used throughout the process consists of alcohol, 750 Cc.; water, 250 Cc.; that each 1000 Gm. of drug in No. 60 powder is moistened with 400 Cc. of the menstruum; that the moistened powder is allowed to macerate packed in the percolator; that the time for maceration in this and all other percolated fluidextracts is forty-eight hours; that the first 800 Cc. is put aside as reserve portion, and that the weak percolate is to be evaporated at 50° C. or under; that no mention is made of first distilling the alcohol. All these points are mentioned, since they form the basis of difference between this and the other fluidextracts of the same class. These other fluidextracts will be found below, grouped according to menstruum employed, the tabulation including such data as fineness of powder, amount of menstruum used for moistening the amount collected as reserve portion, and temperature for evaporation.

The pharmacopœia specifically directs distilling off the alcohol from the weak percolate only in seven cases, and as these have already been enumerated, it seems unnecessary to repeat them in the table.

OTHER OFFICIAL FLUIDEXTRACTS MADE LIKE FLUIDEXTRACT OF ACONITE

Menstruum.	Name.	Fineness of powder.	Moisten drug with—	Reserved portion.	Maximum heat for.
Alcohol (U. S. P.).	Aromatic	U.S.P.	350 Cc.	850 Cc.	50°
	Capsicum	No. 50	500 Cc.	900 Cc.	No mention.
	Cimicifuga	No. 60	250 Cc.	900 Cc.	" "
	Cubeb	No. 40	200 Cc.	900 Cc.	" "
	Gelsemium	No. 60	300 Cc.	900 Cc.	" "
	Ginger	No. 50	250 Cc.	900 Cc.	50°
	Indian cannabis	No. 30	300 Cc.	900 Cc.	Water-bath.
	Savine	No. 40	250 Cc.	900 Cc.	50°
	Veratrum	No. 60	300 Cc.	900 Cc.	50°
Alcohol, 800 Cc.					
Water, 200 Cc.	Belladonna root	No. 60	350 Cc.	800 Cc.	50°
	Eriodictyon	No. 60	400 Cc.	900 Cc.	50°
	Euonymus	No. 40	350 Cc.	800 Cc.	No mention.
	Mezereum	No. 30	400 Cc.	900 Cc.	Water-bath.
	Podophyllum	No. 40	300 Cc.	850 Cc.	" "
	Rhubarb	No. 30	400 Cc.	750 Cc.	70°
	Scopola	No. 40	350 Cc.	800 Cc.	50°
	Serpentaria	No. 50	300 Cc.	900 Cc.	50°
	Staphisagria	No. 40	300 Cc.	800 Cc.	50°
Alcohol, 750 Cc.					
Water, 250 Cc.	Buchu	No. 60	400 Cc.	850 Cc.	50°
	Calamus	No. 40	350 Cc.	900 Cc.	50°
	Eucalyptus	No. 40	400 Cc.	900 Cc.	50°
	Grindelia	No. 30	300 Cc.	850 Cc.	Water-bath.
	Ipecac	No. 80	350 Cc.	850 Cc.	50°
	Leptandra	No. 60	400 Cc.	850 Cc.	No mention.
	Matico	No. 40	300 Cc.	850 Cc.	" "
	Sumbul	No. 30	400 Cc.	850 Cc.	" "
	Valerian	No. 40	300 Cc.	850 Cc.	50°
	Xanthoxylum	No. 40	250 Cc.	900 Cc.	No mention.
Alcohol, 700 Cc.					
Water, 300 Cc.	Calumba	No. 20	300 Cc.	700 Cc.	Water-bath.
Alcohol, 650 Cc.					
Water, 350 Cc.	Convallaria	No. 60	400 Cc.	800 Cc.	No mention.

<i>Menstruum.</i>	<i>Name.</i>	<i>Fineness of powder.</i>	<i>Moisten drug with—</i>	<i>Reserved portion.</i>	<i>Maximum heat for.</i>
Alcohol, 600 Cc.					
Water, 300 Cc.	Bitter orange.....	No. 40	350 Cc.	800 Cc.	50°
	Colchicum seed.....	No. 50	300 Cc.	750 Cc.	No mention.
	Hyoseyamus.....	No. 60	400 Cc.	800 Cc.	50°
	Stramonium.....	No. 40	400 Cc.	800 Cc.	50°
	Viburnum opulus.....	No. 40	300 Cc.	850 Cc.	No mention.
	Viburnum prunifolium.....	No. 40	300 Cc.	850 Cc.	" "
Diluted alcohol	Berberis.....	No. 40	400 Cc.	700 Cc.	" "
(U. S. P.).	Chimaphila.....	No. 30	400 Cc.	800 Cc.	" "
	Chirata.....	No. 30	350 Cc.	850 Cc.	" "
	Coca.....	No. 40	450 Cc.	700 Cc.	50°
	Cypripedium.....	No. 60	350 Cc.	850 Cc.	No mention.
	Digitalis.....	No. 60	400 Cc.	850 Cc.	50°
	Eupatorium.....	No. 40	400 Cc.	800 Cc.	No mention.
	Gentian.....	No. 30	350 Cc.	800 Cc.	" "
	Guarana.....	No. 60	200 Cc.	700 Cc.	" "
	Krameria.....	No. 40	400 Cc.	800 Cc.	" "
	Lappa.....	No. 60	400 Cc.	800 Cc.	" "
	Phytolacca.....	No. 40	400 Cc.	800 Cc.	" "
	Pilocarpus.....	No. 40	350 Cc.	750 Cc.	50°
	Quillaja.....	No. 40	400 Cc.	800 Cc.	No mention.
	Rubus.....	No. 40	350 Cc.	800 Cc.	Water-bath.
	Scutellaria.....	No. 40	350 Cc.	800 Cc.	No mention.
	Spigelia.....	No. 40	300 Cc.	850 Cc.	" "
	Stillingia.....	No. 40	300 Cc.	850 Cc.	" "
Alcohol, 400 Cc.					
Water, 600 Cc.	Cascara sagrada.....	No. 40	400 Cc.	800 Cc.	" "
Alcohol, 500 Cc.					
Water, 800 Cc.	Frangula.....	No. 40	350 Cc.	800 Cc.	" "
Alcohol, 300 Cc.					
Water, 600 Cc.	Quassia.....	No. 40	400 Cc.	900 Cc.	" "
	Sarsaparilla.....	No. 30	400 Cc.	800 Cc.	" "

II. FLUIDEXTRACTS MADE WITH HYDRO-ALCOHOLIC, GLYCERINIC MENSTRA

FLUIDEXTRACTUM APOCYNII. Fluidextract of Apocynum.

Recipe.—Apocynum, in No. 60 powder, one thousand grammes 1000 Gm.
 Glycerin, one hundred cubic centimeters..... 100 Cc.
 Alcohol,
 Water, each, a sufficient quantity,
 To make one thousand centimeters.. 1000 Cc.

Mix the Glycerin with *six hundred cubic centimeters* of Alcohol and *three hundred cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding, first, the remainder of the menstruum, and afterward a mixture of Alcohol and Water, made in the proportion of *six hundred cubic centimeters* of Alcohol to *four hundred cubic centimeters* of Water, until the Apocynum is exhausted. Reserve the first *nine hundred cubic centimeters* of the percolate, and evaporate the remainder, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, and add enough menstruum to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—The fluidextracts made with menstrea containing glycerin are placed in a class separate from those made with purely hydro-alcoholic menstrea, because there is a slight difference in the method of manipulation. As seen in the above typical recipe, in

making 1000 Cc. of the fluidextract, only the first 1000 Cc. of menstruum contains glycerin; the rest of the menstruum required being merely alcohol and water. This necessitates, in the table given below, two columns for menstrea, the column headed "final menstruum" being that employed after the first 1000 Cc. of menstruum (if 1000 Gm. of drug are being percolated) are used up. Bear in mind that if repercolation is used, the first (glycerinic) menstruum is used through the entire operation.

OTHER OFFICIAL FLUIDEXTRACTS MADE LIKE FLUIDEXTRACT OF APOCYNUM

<i>Menstruum.</i>	<i>Name.</i>	<i>Fine- ness of powder.</i>	<i>Moisten drug with—</i>	<i>Reserved portion.</i>	<i>Maximum temperature.</i>	<i>Final menstruum.</i>
Alcohol, 800 Cc.; Water, 100 Cc.; Glycerin, 100 Cc. }	Cinchona.....	No. 60	350 Cc.	700 Cc.	No mention.	Alcohol, 800 Cc.; Water, 200 Cc.
Alcohol, 600 Cc.; Water, 300 Cc.; Glycerin, 100 Cc. }	Geranium.....	No. 30	350 Cc.	800 Cc.	No mention.	Alcohol, 600 Cc.; Water, 400 Cc.
	Hydrastis	No. 60	300 Cc.	750 Cc.	No mention.	Alcohol, 600 Cc.; Water, 300 Cc.
	Pareira	No. 40	400 Cc.	850 Cc.	No mention.	Alcohol, 600 Cc.; Water, 400 Cc.
Diluted alcohol, 900 Cc.; Glycerin, 100 Cc. }	Pomegranate..	No. 30	400 Cc.	800 Cc.	No mention.	Diluted alcohol.
	Quercus	No. 40	400 Cc.	700 Cc.	No mention.	Diluted alcohol.
	Rhus glabra....	No. 40	350 Cc.	800 Cc.	No mention.	Diluted alcohol.
	Rose	No. 20	400 Cc.	750 Cc.	50°	Diluted alcohol.
Alcohol, 300 Cc.; Water, 600 Cc.; Glycerin, 100 Cc. }	Hamamelis leaves.....	No. 40	350 Cc.	850 Cc.	No mention.	Alcohol, 300 Cc.; Water, 600 Cc.
Alcohol, 200 Cc.; Water, 500 Cc.; Glycerin, 300 Cc. }	Uva ursi	No. 30	400 Cc.	800 Cc.	50°	Alcohol, 200 Cc.; Water, 500 Cc.

III. FLUIDEXTRACTS MADE WITH HYDRO-ALCOHOLIC ACID MENSTREA

FLUIDEXTRACTUM CONII. Fluidextract of Conium.

Recipe.—Conium, in No. 40 powder, one thousand grammes.. 1000 Gm.
Acetic Acid, twenty cubic centimeters..... 20 Cc.
Diluted Alcohol, a sufficient quantity,
To make about one thousand cubic centimeters.. 1000 Cc.

Mix the Acetic Acid with *nine hundred and eighty cubic centimeters* of Diluted Alcohol, and, having moistened the powder with *three hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding, first, the remainder of the menstruum, and then Diluted Alcohol, until the Conium is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder, in a porcelain dish, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, and mix thoroughly.

Assay *ten cubic centimeters* of this liquid; from the results thus obtained, ascertain by calculation the amount of coniine in the remainder of the liquid, and add to this enough Diluted Alcohol to make each *one hundred cubic centimeters* of the Fluid-extract contain 0.45 Gm. of coniine.

Remarks.—The statement given under Fluidextract of Apocynum regarding the two menstrea applies with equal force to fluidextracts of this class. The value of the acid constituent is to fix and hold in solution the volatile alkaloid characteristic to the drug. For assay, see Chapter LV.

FLUIDEXTRACTUM ERGOTÆ. Fluidextract of Ergot.

This is made by a process identical with that of fluidextract of conium, except that the drug is in a No. 60 powder; that the first 850 Cc. of percolate are set aside as reserve portion instead of 800 Cc., as in conium; and that no assay is provided.

FLUIDEXTRACTUM NUCIS VOMICÆ. Fluidextract of Nux Vomica.

Recipe.—Nux Vomica, in No. 40 powder, one thousand grammes..... 1000 Gm.
 Acetic Acid, fifty cubic centimeters..... 50 Cc.
 Alcohol,
 Water, each, a sufficient quantity,
 To make about one thousand cubic centimeters.. 1000 Cc.

Mix Alcohol and Water in the proportion of seven hundred and fifty cubic centimeters of Alcohol and two hundred and fifty cubic centimeters of Water. Moisten the powder with one thousand cubic centimeters of the mixture, to which the Acetic Acid had previously been added, and let it digest, in a well-covered vessel, in a warm place, during forty-eight hours. Then pack it in a cylindrical glass percolator, gradually pour menstruum upon it, and allow the percolation to proceed slowly until the Nux Vomica is practically exhausted. Reserve the first nine hundred cubic centimeters of the percolate, distil off the Alcohol from the remainder by means of a water-bath, and evaporate the residue, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, and mix thoroughly.

Assay ten cubic centimeters of this liquid; from the results thus obtained ascertain by calculation the amount of strychnine in the remainder of the liquid; add to this enough menstruum to make each one hundred cubic centimeters of the finished Fluidextract contain 1 Gm. of strychnine.

Remarks.—Note that the recipe for this fluidextract differs from the two just given only in a few details; that the alcoholic strength of the menstruum is greater, and that the drug is digested with a large amount of menstruum before percolation. For assay, see Chapter LV.

IV. FLUIDEXTRACTS MADE WITH HYDRO-ALCOHOLIC ALKALINE MENSTRUUA**FLUIDEXTRACTUM SENEGÆ.** Fluidextract of Senega.

Recipe.—Senega, in No. 40 powder, one thousand grammes.... 1000 Gm.
 Solution of Potassium Hydroxide, thirty cubic centimeters..... 30 Cc.
 Alcohol,
 Water, each, a sufficient quantity,
 To make one thousand cubic centimeters.. 1000 Cc.

Mix the Solution of Potassium Hydroxide with six hundred cubic centimeters of Alcohol and three hundred cubic centimeters of Water, and, having moistened the powder with four hundred and fifty cubic centimeters of the mixture, pack it firmly in a cylindrical glass percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, loose the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding, first, the remainder of the menstruum, and then a mixture of Alcohol and Water, made in the proportion of six hundred cubic centimeters of Alcohol to three hundred cubic centimeters of Water, until the Senega is exhausted. Reserve the first eight hundred and fifty cubic centimeters of the percolate, and evaporate the remainder, in a porcelain dish, to a soft extract; dissolve this in the reserved portion, and add enough of the last-mentioned mixture of Alcohol and Water to make the Fluidextract measure one thousand cubic centimeters.

Remarks.—As mentioned under Syrup of Senega (p. 201), the drug contains pectin, which is liable to separate from liquid preparations in a gelatinous mass. Alkalis prevent this separation of pectin, hence the use of solution of potassium hydroxide in the present pharmacopœial recipe for the fluidextract and the employment of ammonia water in making the syrup according to the process of 1890 (p. 201).

FLUIDEXTRACTUM TARAXACI. Fluidextract of Taraxacum.

Recipe.—Taraxacum, in No. 30 powder, *one thousand grammes*..... 1000 Gm.
 Solution of Sodium Hydroxide, *fifty cubic centimeters*..... 50 Cc.
 Diluted Alcohol, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Moisten the powder with *three hundred cubic centimeters* of Diluted Alcohol, and pack it firmly in a cylindrical percolator; then add enough Diluted Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Diluted Alcohol, until the Taraxacum is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder to a soft extract; dissolve this in the reserved portion, add the Solution of Sodium Hydroxide, and enough Diluted Alcohol to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—Note that this recipe differs from that for making fluidextract of senega in the fact that the alkaline solution is not added to the menstruum, as in senega, but to the finished product. The object of the alkali is similar to the reason for its use in fluidextract of senega—to keep in solution those active principles likely to precipitate.

V. FLUIDEXTRACTS MADE WITH ACETIC ACID MENSTRUUM

FLUIDEXTRACTUM LOBELIÆ. Fluidextract of Lobelia.

Recipe.—Lobelia, in No. 50 powder, *one thousand grammes*.. 1000 Gm.
 Acetic Acid,
 Water, each, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Mix *two hundred and seventy-five cubic centimeters* of Acetic Acid with *seven hundred and twenty-five cubic centimeters* of Water, and, having moistened the powder with *three hundred and fifty cubic centimeters* of the mixture, pack it firmly in a cylindrical glass percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding menstruum, using the same proportions of Acetic Acid and Water as before, until the Lobelia is exhausted. Reserve the first *nine hundred cubic centimeters* of the percolate, and evaporate the remainder, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, and add enough menstruum to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—This line of fluidextracts, for which the name *acettracts* has been suggested, differ from the acetic acid fluidextracts, conium, ergot, and nux vomica, described above, in the fact that while those three are made with alcoholic menstrua containing a little acetic

acid, the ones we are now considering are made with an approximately 10 per cent. acetic acid and contain no alcohol.

The truth of the above statement can be easily proved by figuring out by alligation how much 10 per cent. acetic acid can be made from 275 Cc. of the official 36 per cent. acetic acid directed in the above recipe.

The value of acetic acid as a solvent has been the subject of careful study during recent years, and, as a result, in the last revision three "acetracts"—lobelia, sanguinaria, and squill—were given recognition.

FLUIDEXTRACTUM SANGUINARIÆ. Fluidextract of Sanguinaria.

The official recipe for this extract is like that for fluidextract of lobelia just given, except that the drug must be in a No. 30 powder, that it is to be moistened with 300 Cc. of menstruum, that the first 850 Cc. of percolate is to be put aside as reserve portion, and that no mention is made of the temperature for evaporation of the weak percolate.

Fluidextract of squill, while made with acetic acid menstruum, is prepared without evaporation of the weak percolate, hence is discussed below.

FLUIDEXTRACTS MADE BY EVAPORATION OF HOT PERCOLATED INFUSIONS

This process differs from percolation with partial evaporation in the fact that the entire aqueous percolate is evaporated to certain bulk, and then enough alcohol is added to preserve the finished product from fermentation. But two official fluidextracts are made in this way—glycyrrhiza and tritium.

FLUIDEXTRACTUM GLYCYRRHIZÆ. Fluidextract of Glycyrrhiza.

<i>Recipe.</i> —Glycyrrhiza, in No. 20 powder, <i>one thousand</i>	
<i>grammes</i>	1000 Gm.
Glycerin, <i>two hundred and fifty cubic centimeters</i>	250 Cc.
Ammonia Water, <i>fifty cubic centimeters</i>	50 Cc.
Alcohol,	
Water, each, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Pour *four hundred cubic centimeters* of boiling Water upon the Glycyrrhiza, contained in a suitable vessel, and allow it to stand for one hour. Pack the moistened powder loosely in a metallic percolator, pour boiling Water upon it, and allow the percolation to proceed, supplying boiling Water until the Glycyrrhiza is exhausted. Evaporate the percolate at a moderate heat, until it measures *four hundred and fifty cubic centimeters*, and when cool, add *four hundred and fifty cubic centimeters* of Alcohol, mix well, and set it aside for three days. After filtering the liquid, distil it until *five hundred cubic centimeters* of distillate have been obtained; transfer the liquid in the still to a suitable container, add the Glycerin, Ammonia Water, and *two hundred cubic centimeters* of Alcohol. Finally, add sufficient Water to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—Explaining this rather intricate process, it may be said that the object of adding to the 500 Cc. of evaporated aqueous percolate 500 Cc. of alcohol (only eventually to distil the latter off) is to precipitate the mucilaginous and starchy matter extracted from

the drug with the boiling water, and which would prove a source of precipitation were it allowed to remain in the finished fluidextract. This fluidextract of glycyrrhiza is similar to the popular commercial forms intended "for quinine mixtures," and is much superior to the alcoholic fluidextract of the pharmacopœia of 1890.

FLUIDEXTRACTUM TRITICI. Fluidextract of Triticum.

Recipe.—Triticum, finely cut, one thousand grammes. 1000 Gm.

Alcohol,

Water, each, a sufficient quantity,

To make one thousand cubic centimeters. . . 1000 Cc.

Pack the Triticum in a cylindrical metallic percolator, pour boiling Water upon it, and allow the percolation to proceed, supplying boiling water, as required, until the Triticum is exhausted. Evaporate the percolate to *seven hundred and fifty cubic centimeters*, and having added to it *two hundred and fifty cubic centimeters* of Alcohol, mix well and set it aside for forty-eight hours. Then filter the liquid and add to the filtrate enough of a mixture of Alcohol and Water made in the proportion of *one volume* of Alcohol to *three volumes* of Water to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—The fluidextract of triticum is interesting because made by evaporation of an infusion of triticum with boiling water, to a suitable bulk, and adding thereto 25 per cent. of alcohol, the object of the alcohol being to prevent fermentation, as well as to precipitate starchy and mucilaginous matter.

FLUIDEXTRACTS MADE BY PERCOLATION WITHOUT EVAPORATION

In the case of two official fluidextracts—squill and wild cherry—the drug is so readily exhausted that the first 1000 Cc. percolate (*if the percolation is carefully conducted*) will contain practically all the active constituents of 1000 Gm. of drug. In these cases, therefore, the percolation is discontinued when 1000 Cc. of percolate are obtained. Too great stress cannot be laid, however, on the necessity for *careful percolation*, and this gives the process the same weakness already outlined when discussing percolation with incomplete exhaustion (p. 242).

FLUIDEXTRACTUM SCILLÆ. Fluidextract of Squill.

Recipe.—Squill, in No. 20 powder, one thousand grammes. . . 1000 Gm.

Acetic Acid,

Water, each, a sufficient quantity,

To make one thousand cubic centimeters. . . 1000 Cc.

Mix *two hundred and seventy-five cubic centimeters* of Acetic Acid with *seven hundred and twenty-five cubic centimeters* of Water, and, having added *eight hundred cubic centimeters* of the mixture to the powder, allow it to macerate, in a well-covered glass or porcelain vessel, during forty-eight hours, then transfer it to a conical glass percolator, and allow the percolation to proceed slowly, adding menstruum gradually, using the same proportions of Acetic Acid and Water as before, until the Fluidextract measures *one thousand cubic centimeters*.

Remarks.—This recipe differs from that for the fluidextracts of lobelia and sanguinaria in directing that 1000 Gm. of drug be macerated with 800 Cc. of the acetic acid menstruum (representing about 10 per

cent. absolute acetic acid) before packing, and that the percolation be conducted so that when 1000 Cc. of percolate are obtained, the drug will be exhausted.

FLUIDEXTRACTUM PRUNI VIRGINIANÆ. Fluidextract of Wild Cherry.

Recipe.—Wild Cherry, in No. 30 powder, *one thousand grammes*..... 1000 Gm.
 Glycerin, *two hundred cubic centimeters*..... 200 Cc.
 Alcohol,
 Water, each, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Mix the Glycerin with *two hundred cubic centimeters* of Alcohol and *six hundred cubic centimeters* of Water, and, having moistened the powder with *three hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical glass percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours; then allow the percolation to proceed, gradually adding, first, the remainder of the menstruum, and afterward a mixture of Alcohol and Water, made in the proportion of *two hundred cubic centimeters* of Alcohol to *eight hundred cubic centimeters* of Water, and allow the percolation to proceed very slowly, until the Fluidextract measures *one thousand cubic centimeters*.

Remarks.—In this recipe the drug is percolated with a menstruum of alcohol, water, and glycerin, with such care that when 1000 Cc. of percolate have passed from 1000 Gm. of drug, the latter will be exhausted. The chief object of this requirement is to avoid heat, to which the constituents of wild cherry are very sensitive.

SPECIAL FLUIDEXTRACTS

Under this heading are grouped the few remaining official fluidextracts which either do not lend themselves to the grouping followed on the preceding pages, or possess some striking peculiarity worthy of special mention.

FLUIDEXTRACTUM LUPULINI. Fluidextract of Lupulin.

Recipe.—Lupulin, *one thousand grammes*..... 1000 Gm.
 Alcohol, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. 1000 Cc.

Pack the Lupulin firmly in a cylindrical percolator; then add enough Alcohol to saturate the Lupulin and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Alcohol, until the Lupulin is exhausted. Reserve the first *nine hundred cubic centimeters* of the percolate, and evaporate the remainder to a soft extract; dissolve this in the reserved portion, and add enough Alcohol to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—The only difference between the manufacture of this fluidextract and the others made by percolation with partial evaporation is that the drug is packed into the percolator without previous moistening.

FLUIDEXTRACTUM RHAMNI PURSHIANÆ AROMATICUM. Aromatic Fluidextract of Cascara Sagrada.

<i>Recipe.</i> —Cascara Sagrada, in No. 40 powder, one thousand grammes.....		1000 Gm.
Glycyrrhiza, in No. 30 powder, one hundred grammes.....		100 Gm.
Magnesium Oxide, one hundred and twenty-five grammes.....		125 Gm.
Glycerin, two hundred and fifty cubic centimeters....		250 Cc.
Compound Spirit of Orange, ten cubic centimeters..		10 Cc.
Alcohol,		
Diluted Alcohol,		
Water, each, a sufficient quantity,		
To make one thousand cubic centimeters..		1000 Cc.

Mix the Cascara Sagrada, Glycyrrhiza, and Magnesium Oxide thoroughly, and, having added *two thousand cubic centimeters* of Water, allow the mixture to macerate for twelve hours, and then dry it at a gentle heat. Mix the Glycerin with *five hundred cubic centimeters* of Alcohol and *two hundred and fifty cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding, first the remainder of the menstruum, and afterward Diluted Alcohol, until the powder is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder to a soft extract; dissolve this in the reserved portion, and add the Compound Spirit of Orange and enough Diluted Alcohol to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—This fluidextract is made by percolation with partial evaporation, all general details being like the other fluidextracts of this class. The important distinction is the treatment of the drug, before percolating, with magnesium oxide, this being done to remove most of the bitter taste from the cascara sagrada (p. 870). Note, of course, the constituents added to make the product true to its name, “aromatic,” these constituents being glycyrrhiza and compound spirit of orange.

FLUIDEXTRACTUM SARSAPARILLÆ COMPOSITUM. Compound Fluidextract of Sarsaparilla.

<i>Recipe.</i> —Sarsaparilla, in No. 30 powder, seven hundred and fifty grammes.....		750 Gm.
Glycyrrhiza, in No. 30 powder, one hundred and twenty grammes.....		120 Gm.
Sassafras, in No. 30 powder, one hundred grammes..		100 Gm.
Mezereum, in No. 30 powder, thirty grammes.....		30 Gm.
Glycerin, one hundred cubic centimeters.....		100 Cc.
Diluted Alcohol, a sufficient quantity,		
To make about one thousand cubic centimeters..		1000 Cc.

Mix the Glycerin with *nine hundred cubic centimeters* of Diluted Alcohol, and, having moistened the mixed powders with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough Diluted Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Diluted Alcohol until the powder is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder to a soft extract; dissolve this in the reserved portion, and add enough Diluted Alcohol to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—This preparation belongs to those fluidextracts prepared by percolation and partial evaporation, and the only reason it is grouped in the special class is because it is the only official “compound” fluidextract. Note carefully the ingredients, and it must be borne in mind that it does not contain the same constituents as the compound syrup of sarsaparilla, and it must in no instance, therefore, be used in preparing the official compound syrup.

FLUIDEXTRACTUM SENNÆ. Fluidextract of Senna.

Recipe.—Senna, in No. 40 powder, *one thousand grammes*. . . **1000 Gm.**
Alcohol,

Diluted Alcohol, each, *a sufficient quantity*,

To make *one thousand cubic centimeters*. . . **1000 Cc.**

Moisten the powder with *three hundred and fifty cubic centimeters* of Alcohol, pack it firmly in a cylindrical percolator, and percolate it with Alcohol until the Senna is exhausted. The alcoholic percolate thus obtained is to be rejected. Remove the powder from the percolator, dry it, and, having moistened it with *four hundred cubic centimeters* of Diluted Alcohol, pack it firmly in a cylindrical percolator; then add enough Diluted Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Diluted Alcohol until the Senna is exhausted. Reserve the first *eight hundred cubic centimeters* of the percolate, and evaporate the remainder, at a temperature not exceeding 50° C. (122° F.), to a soft extract; dissolve this in the reserved portion, and add enough Diluted Alcohol to make the Fluidextract measure *one thousand cubic centimeters*.

Remarks.—The recipe for this fluidextract is peculiar, in that it directs that the senna be exhausted with alcohol before percolating with the regular menstruum—diluted alcohol. As mentioned on p. 871, senna contains a griping principle, and this, as well as most of the odor and taste, is removed by treatment with alcohol without loss of the valuable purgative principles.

The recipe of the present pharmacopœia, given above, is practically identical with that for deodorized fluidextract of senna of the former edition of the National Formulary.

DOSES OF THE OFFICIAL FLUIDEXTRACTS

0.05 Cc. (1 minim).....	Fluidextracts of aconite, belladonna root, capsicum, digitalis, gelsemium, Indian cannabis, ipecac (as expectorant), nux vomica, scopola, staphisagria, and stramonium.
0.10 Cc. (1½ minims).....	Fluidextracts of phytolacca (as alternative), sanguinaria, squill, and veratrum.
0.20 Cc. (3 minims).....	Fluidextracts of colchicum seed, conium, hyoscyamus, and quillaja.
0.30 Cc. (5 minims).....	Fluidextract of savine.
0.50 Cc. (8 minims).....	Fluidextracts of convallaria, euonymus, lobelia, lupulin, podophyllum, and quassia.
1.00 Cc. (15 minims).....	Fluidextracts of apocynum, aromatic, bitter orange peel, calamus, cascara sagrada, cascara sagrada aromatic, chirata, cimicifuga, cinchona, cubeb, cypripedium, eriodictyon, frangula, gentian, geranium, ginger, krameria, quercus, rhubarb, rhus glabra, rubus, scutellaria, senega, and serpentaria.

Doses of the Official Fluidextracts—(Continued)

2.00 Cc. (30 minims).....	Fluidextracts of berberis, buchu, calumba, chimaphila, coca, ergot, eucalyptus, eupatorium, glycyrrhiza, grindelia, guarana, hamamelis leaves, hydrastis, lappa, leptandra, pareira, pilocarpus, pomegranate, rose, sarsaparilla, sarsaparilla compound, senna, stillingia, sumbul, uva ursi, valerian, viburnum opulus, viburnum prunifolium, xanthoxylum, and wild cherry.
4.00 Cc. (1 fluidrachm).....	Fluidextracts of matico and spigelia.
8.00 Cc. (2 fluidrachms).....	Fluidextracts of taraxacum and triticum.
No dose given by pharmacopœia....	Fluidextract of mezereum.

Official Fluidextracts Directed to be Assayed

(Details in Chapter LV.)

<i>Fluidextract.</i>	<i>Assayed Strength.</i>
Aconite.....	0.4 per cent. aconitine.
Belladonna root.....	0.4 per cent. mydriatic alkaloids from belladonna.
Cinchona.....	4.0 per cent. ether-soluble alkaloids from cinchona.
Coca.....	0.5 per cent. ether-soluble alkaloids from coca.
Colchicum seed.....	0.4 per cent. colchicine.
Conium.....	0.45 per cent. coniine.
Guarana.....	3.5 per cent. alkaloids from guarana.
Hydrastis.....	2.0 per cent. hydrastine.
Hyoseyamus.....	0.075 per cent. mydriatic alkaloids from hyoseyamus.
Ipecac.....	1.5 per cent. alkaloids from ipecac.
Nux vomica.....	1.0 per cent. strychnine.
Pilocarpus.....	0.4 per cent. alkaloids from pilocarpus.
Scopola.....	0.5 per cent. mydriatic alkaloids from scopola.
Stramonium.....	0.25 per cent. mydriatic alkaloids from stramonium.

WINES

Wines are alcoholic liquids prepared from drugs by the process of solution, maceration, or percolation, differing from tinctures only in the solvent employed—wine being used instead of various strengths of alcohol.

In the pharmacopœia of 1890 white wine was used as a solvent to the exclusion of red wine, but in the present pharmacopœia one official wine (*Vinum Cocæ*) is made with red wine.

Both *red wine* and *white wine* can be more profitably discussed in Part IV., so in this section we will consider only the pharmaceutic wines—those prepared from the two native wines.

TABLE OF OFFICIAL WINES

(ALL GALENIC)

*By solution:**Solvent, white wine.*

Active ingredient a chemical...	{ Wine of antimony. Bitter wine of iron. Wine of iron.
Active ingredient a fluidextract.	{ Wine of colchicum seed. Wine of ergot. Wine of ipecac.

Solvent, red wine.

Active ingredient a fluidextract... Wine of coca.

By maceration..... Wine of opium.

By percolation..... { Wine of colchicum root (U. S. P. 1890).
Wine of ergot (U. S. P. 1890).

SPECIAL NOTES ON OFFICIAL WINES

VINUM ANTIMONII. Wine of Antimony.

<i>Recipe.</i> —Antimony and Potassium Tartrate, four grammes..	4 Gm.
Boiling Distilled Water, sixty-five cubic centimeters.....	65 Cc.
Alcohol, one hundred and seventy-five cubic centimeters.....	175 Cc.
White Wine, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Antimony and Potassium Tartrate in the Boiling Distilled Water. Add this solution to a mixture of the Alcohol with *seven hundred and twenty-five cubic centimeters* of White Wine; mix well, and allow the mixture to stand until it has cooled. Then filter and add sufficient White Wine through the filter to make the liquid measure *one thousand cubic centimeters*.

Remarks.—This wine contains $\frac{4}{10}$ of 1 per cent. of tartar emetic, and is a constituent of *Brown Mixture* (p. 265).

Dose.—1 Cc. (15 minims).

VINUM COCÆ. Wine of Coca.

<i>Recipe.</i> —Fluidextract of Coca, sixty-five cubic centimeters...	65 Cc.
Alcohol, seventy-five cubic centimeters.....	75 Cc.
Sugar, sixty-five grammes.....	65 Gm.
Red Wine, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the sugar in *five hundred cubic centimeters* of Red Wine, add the Alcohol and Fluidextract of Coca, and enough Red Wine to make the liquid measure *one thousand cubic centimeters*. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—This new official is intended to replace the many proprietary wines of coca now enjoying so much popularity. While the pharmacopœia directs use of official “red wine,” which includes a large number of commercial varieties of wine, the careful pharmacist will employ a good grade of claret wine.

Dose.—16 Cc. (4 fluidrachms).

VINUM COLCHICI SEMINIS. Wine of Colchicum Seed.

<i>Recipe.</i> —Fluidextract of Colchicum Seed, one hundred cubic centimeters.....	100 Cc.
Alcohol, one hundred and fifty cubic centimeters.....	150 Cc.
White Wine, seven hundred and fifty cubic centimeters.....	750 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—The strength of this preparation was reduced in the last revision. The wine of the pharmacopœia of 1890 was made by maceration, and represented 15 per cent. of the ground drug. The present official wine is made from the fluidextract, and represents 10 per cent. of the drug.

Dose.—2 Cc. (30 minims).

VINUM COLCHICI RADICIS (U. S. P. 1890). Wine of Colchicum Root.

Recipe.—This former official is made by maceration of the ground corm of colchicum with white wine, fortified by addition of alcohol, and representing 40 per cent. of the drug.

VINUM ERGOTÆ. Wine of Ergot.

<i>Recipe.</i> —Fluidextract of Ergot, two hundred cubic centimeters	200 Cc.
Alcohol, fifty cubic centimeters	50 Cc.
White Wine, seven hundred and fifty cubic centimeters	750 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—Made by simple solution from the fluidextract, like wine of colchicum seed, but contains more fluidextract (20 per cent.) and less alcohol.

Dose.—8 Cc. (2 fluidrachms).

VINUM FERRI. Wine of Iron.

<i>Recipe.</i> —Iron and Ammonium Citrate, forty grammes	40 Gm.
Tincture of Sweet Orange Peel, sixty cubic centimeters	60 Cc.
Syrup, one hundred cubic centimeters	100 Cc.
White Wine, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Iron and Ammonium Citrate in seven hundred cubic centimeters of White Wine. Add to this the Tincture of Sweet Orange Peel and the Syrup, and, lastly, enough White Wine to make the product measure one thousand cubic centimeters. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—This consists of 4 per cent. iron and ammonium citrate dissolved in white wine, flavored with tincture of sweet orange peel, and sweetened with syrup.

Dose.—8 Cc. (2 fluidrachms).

VINUM FERRI AMARUM. Bitter Wine of Iron.

<i>Recipe.</i> —Soluble Iron and Quinine Citrate, fifty grammes...	50 Gm.
Tincture of Sweet Orange Peel, sixty cubic centimeters	60 Cc.
Syrup, three hundred cubic centimeters	300 Cc.
White Wine, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Soluble Iron and Quinine Citrate in five hundred cubic centimeters of White Wine. Add to this the Tincture of Sweet Orange Peel and the Syrup, and, lastly, enough White Wine to make the product measure one thousand cubic centimeters. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—This is substantially wine of iron made bitter by addition of quinine. It contains 5 per cent. soluble iron and quinine citrate, representing almost $\frac{6}{10}$ of 1 per cent. of quinine.

Dose.—8 Cc. (2 fluidrachms).

VINUM IPECACUANHÆ. Wine of Ipecac.

Recipe.—Fluidextract of Ipecac, one hundred cubic centimeters 100 Cc.
 Alcohol, one hundred cubic centimeters..... 100 Cc.
 White Wine, eight hundred cubic centimeters..... 800 Cc.
 To make one thousand cubic centimeters.. 1000 Cc.

Mix them. Set the mixture aside for two days; then filter through paper, in a well-covered funnel.

Remarks.—This wine is made from the fluidextract, like wine of colchicum seed, and, like it, represents 10 per cent. of the drug.

Dose.—1 Cc. (15 minims).

VINUM OPII. Wine of Opium.

Recipe.—Granulated Opium, one hundred grammes..... 100 Gm.
 Saigon Cinnamon, in No. 60 powder, ten grammes.. 10 Gm.
 Cloves, in No. 30 powder, ten grammes. 10 Gm.
 Alcohol,
 White Wine, each, a sufficient quantity,
 To make one thousand cubic centimeters.. 1000 Cc.

Mix one hundred and fifty cubic centimeters of Alcohol and eight hundred and fifty cubic centimeters of White Wine. Macerate the Opium, Saigon Cinnamon, and Cloves in a stoppered container, in a moderately warm place, with seven hundred and fifty cubic centimeters of this menstruum, during seven days, with occasional agitation; then filter through purified cotton, in a well-covered funnel, returning the first portions until the filtrate passes perfectly clear, and finally pass enough menstruum through the residue to make the liquid measure one thousand cubic centimeters.

Remarks.—Wine of opium, sometimes called “Sydenham’s laudanum,” is made by macerating granulated opium, Saigon cinnamon, and cloves with white wine, fortified with 15 per cent. of alcohol. Since it represents 10 per cent. of opium, and since the opium, in turn, contains 12 to 12½ per cent. of morphine, the wine of opium should contain 1.2 to 1.25 per cent. of morphine.

Dose.—0.5 Cc. (8 minims).

Doses of Official Wines

0.5 Cc. (8 minims).....	Wine of opium.
1.0 Cc. (15 minims).....	Wines of antimony and ipecac.
2.0 Cc. (30 minims).....	Wine of colchicum seed.
8.0 Cc. (2 fluidrachms).....	Wines of ergot, iron, and iron bitter.
16.0 Cc. (4 fluidrachms).....	Wine of coca.

OLEORESINS

The pharmaceutic oleoresins are liquid preparations of drugs, containing volatile oil and resin, obtained by percolation of such drug with acetone, ether, or alcohol, and subsequent distillation of the solvent from the dissolved oleoresins.

There are two classes of oleoresins—the *natural oleoresins* and the *pharmaceutic oleoresins*. The former are mixtures of volatile oil and resin exuding from plants, and, as example of these, may be cited the drugs turpentine and copaiba, which are discussed in

Chapter XLVII. These are quite different from the pharmaceutic oleoresins defined above as being made by percolation with the appropriate solvent.

It is self-evident that we can prepare pharmaceutic oleoresins only from such drugs as contain volatile oil and resin; that such preparations as "oleoresin of cinchona" and "oleoresin of gentian" are impossible, since neither drug contains oil and resin in appreciable quantities. As drugs containing oil and resin are not very abundant, the number of oleoresins are limited, only six being official.

Manufacture.—The pharmaceutic oleoresins are manufactured by placing the drug *without moistening* in a suitable apparatus, and percolating with the appropriate solvent. When the solvent is distilled from the percolate, there is a small quantity of oily liquid left behind, and this constitutes the official oleoresin.

If the menstruum used in percolation is alcohol, the extraction is carried on in an ordinary percolator; but if the solvent is acetone or ether, extraction in an ordinary percolator is both wasteful and dangerous, as far as ether is concerned, by reason of the inflammability of the solvent.

Ether being so volatile, percolation must be conducted in apparatus so arranged to reduce evaporation to the minimum. Such a percolator is pictured in Fig. 165. Of course, both percolator and receiving bottle cannot be tightly corked, else the dropping of the liquid would be impossible. Accordingly, the corks closing the tops of the percolator and receiving bottle are pierced by glass tubes drawn to tapering points, through which the air may pass without very great evaporation of the ether.

It is found convenient to connect these glass tubes with a rubber tube, although some sticklers disapprove of the use of the rubber tubing by reason of the contamination, through possible solution, of a small part of the rubber by the vapors of ether as they pass through.

Strength.—The strength of oleoresins is uncertain, inasmuch as different samples of the same drug may contain entirely different amounts of their oleoresinous constituent. The oleoresins, however, represent the most concentrated form of liquid pharmaceuticals, being usually from five to ten times stronger than the drug.

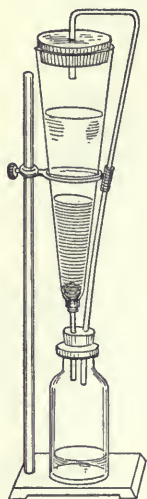


Fig. 165.—Volatile liquid percolator.

TABLE OF OFFICIAL OLEORESINS (ALL GALENIC)

Solvent.

<i>Acetone</i>	{	Oleoresin of aspidium.
		Oleoresin of capsicum.
		Oleoresin of ginger.
		Oleoresin of lupulin.
		Oleoresin of pepper.
<i>Alcohol</i>		Oleoresin of cubeb.

As already mentioned, a marked difference between the present pharmacopœial recipe for oleoresins and that of the pharmacopœia of 1890 is the employment of acetone and alcohol as solvents instead of ether. The change is an improvement, since the use of ether was both costly and dangerous.

SPECIAL NOTES ON OFFICIAL OLEORESINS

OLEORESINA ASPIDII. Oleoresin of Aspidium.

Recipe.—**Aspidium**, recently reduced to No. 40 powder, *five hundred grammes*. 500 Gm.
Acetone, a sufficient quantity.

Introduce the Aspidium into a cylindrical glass percolator, provided with a stop-cock, and arranged with a cover and a receptacle suitable for volatile liquids. Pack the powder firmly, and percolate slowly with Acetone, added in successive portions, until the Aspidium is exhausted. Recover the greater part of the Acetone from the percolate by distillation on a water-bath, and, having transferred the residue to a dish, allow the remaining Acetone to evaporate spontaneously in a warm place. Keep the Oleoresin in a well-stoppered bottle.

Note.—Oleoresin of Aspidium usually deposits, on standing, a granular, crystalline substance. This should be thoroughly mixed with the liquid portion before use.

Remarks.—The precipitate that forms in this oleoresin consists of the valuable constituent, flicic acid, and this oleoresin must, therefore, be dispensed with a “shake” label.

Note that the drug used must be freshly ground.

Dose.—2 Gm. (30 grains).

OLEORESINA CAPSICI. Oleoresin of Capsicum.

Recipe.—**Capsicum**, in No. 40 powder, *five hundred grammes*. . 500 Gm.
Acetone, a sufficient quantity.

Introduce the Capsicum into a cylindrical glass percolator, provided with a stop-cock, and arranged with a cover and a receptacle suitable for volatile liquids. Pack the powder firmly, and percolate slowly with Acetone, added in successive portions, until *eight hundred cubic centimeters* of percolate have been obtained. Recover the greater part of the Acetone from the percolate by distillation on a water-bath, and, having transferred the residue to a dish, allow the remaining Acetone to evaporate spontaneously in a warm place. Then pour off the liquid portion, transfer the remainder to a glass funnel provided with a pledget of cotton, and when the separated fatty matter (which is to be rejected) has been completely drained, mix the liquid portions together. Keep the Oleoresin in a well-stoppered bottle.

Remarks.—Oleoresin of capsicum is made by percolating the drug with acetone and recovering the greater portion of this solvent by distillation of the percolate on the water-bath, the last traces being permitted to evaporate spontaneously. The residue, after evaporation of the acetone, consists of the oleoresins plus a certain quantity of inert fatty substance, which is separated from the valuable liquid by straining through cotton. The oleoresin of capsicum is used in the preparation of capsicum plaster.

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

OLEORESINA CUBEÆ. Oleoresin of Cubeb.

Recipe.—**Cubeb**, in No. 30 powder, *five hundred grammes*. . . 500 Gm.
Alcohol, a sufficient quantity.

Introduce the Cubeb into a cylindrical glass percolator, pack the powder firmly, and percolate slowly with Alcohol, added in successive portions, until the Cubeb is exhausted. Recover the greater part of the Alcohol from the percolate by distillation on a water-bath, and, having transferred the residue to a dish, allow the remaining Alcohol to evaporate, with constant stirring, in a warm place. Keep the Oleoresin in a well-stoppered bottle.

Note.—Oleoresin of Cubeb deposits, after standing for some time, a waxy and crystalline matter, which should be rejected, the liquid portion only being used.

Remarks.—Note that this is the only official oleoresin made by percolating a drug with alcohol. Note that the waxy precipitate is to be rejected, since it is inert and not valuable, as is the precipitate in oleoresin of aspidium. This oleoresin is a constituent of the official *troches of cubeb*.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

OLEORESINA LUPULINI. Oleoresin of Lupulin.

Recipe.—The same as for making oleoresin of aspidium, except that the unground lupulin glands are employed, and that the powder is packed very lightly.

Dose.—200 milligrammes (3 grains).

OLEORESINA PIPERIS. Oleoresin of Pepper.

Recipe.—Pepper, in No. 40 powder, five hundred grammes... 500 Gm.

Acetone, a sufficient quantity.

Introduce the Pepper into a cylindrical glass percolator, provided with a stop-cock, and arranged with a cover and a receptacle for volatile liquids. Pack the powder firmly, and percolate slowly with Acetone, added in successive portions, until the Pepper is exhausted. Recover the greater part of the Acetone from the percolate by distillation on a water-bath, and, having transferred the residue to a dish, set this aside in a warm place until the remaining Acetone has evaporated, and the deposition of crystals of piperin has ceased. Lastly, separate the Oleoresin from the piperin by straining through purified cotton. Keep the Oleoresin in a well-stoppered bottle.

Remarks.—This is prepared like the oleoresin of capsicum. After the acetone has mostly evaporated, the piperin begins to crystallize from the liquid, and the pharmacopœia directs that these crystals of piperin be removed from the oleoresin by straining through cotton.

This oleoresin is largely prescribed in the South as *oil of black pepper*.

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

OLEORESINA ZINGIBERIS. Oleoresin of Ginger.

Recipe.—Made like oleoresin of aspidium, except that the drug is to be in a No. 60 powder and not necessarily freshly ground.

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

Doses of Official Oleoresins

30 milligrammes ($\frac{1}{2}$ grain).....	Oleoresins of capsicum, ginger, and pepper.
200 " (3 grains).....	Oleoresin of lupulin.
500 " ($7\frac{1}{2}$ grains).....	Oleoresin of cubeb.
2 grammes (30 grains).....	Oleoresin of aspidium.

VINEGARS

Vinegars are liquid preparations obtained by extracting the active principle of drugs by maceration or percolation with vinegar or with diluted acetic acid.

This is one of the oldest classes of liquid medicinal preparations, and resembles tinctures in everything but the solvent employed. Diluted acetic acid, or its impure form,—vinegar,—is an excellent solvent for the active principles of many drugs, and is a preservative as well; hence the ancient uses of medicated vinegars and the present employment of 10 per cent. acetic acid as menstruum in some fluid-extracts (p. 251).

However, the vinegars themselves have been largely supplanted by tinctures and fluidextracts, there now being but two retained in the pharmacopœia. The menstruum of both of these is diluted acetic acid; both are prepared by maceration, and both represent 10 per cent. of the active drug. The two official are *vinegar of squill* and *vinegar of opium*. The pharmacopœia of 1880 directed percolation for both, a method poorly adapted to both drugs.

SPECIAL NOTES ON OFFICIAL VINEGARS

ACETUM OPII. Vinegar of Opium.

<i>Recipe</i> .—Powdered Opium, one hundred grammes.....	100 Gm.
Myristica, in No. 30 powder, thirty grammes.....	30 Gm.
Sugar, two hundred grammes.....	200 Gm.
Diluted Acetic Acid, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Macerate the Opium and Myristica in *five hundred cubic centimeters* of Diluted Acetic Acid during seven days, frequently stirring; then strain through muslin of close texture, and express the liquid. Mix the residue with *two hundred cubic centimeters* of Diluted Acetic Acid until a uniform magma is produced, then strain and express again. Mix and filter the strained liquids, dissolve the Sugar in the filtrate, and pass enough Diluted Acetic Acid through the filter to make the product measure *one thousand cubic centimeters*.

Remarks.—This preparation, called *black drop*, was once dispensed with fatal results on a prescription calling for *black draught* (compound infusion of senna).

Dose.—0.5 Cc. (8 minims).

ACETUM SCILLÆ. Vinegar of Squill.

<i>Recipe</i> .—Squill, in No. 20 powder, one hundred grammes....	100 Gm.
Diluted Acetic Acid, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Macerate the Squill with *nine hundred cubic centimeters* of Diluted Acetic Acid during seven days, frequently stirring; then strain through muslin, and wash the mass on the strainer with enough Diluted Acetic Acid to make the strained liquid measure nearly *one thousand cubic centimeters*. Heat this liquid to boiling, filter while hot, and when cooled add sufficient Diluted Acetic Acid to make the product measure *one thousand cubic centimeters*.

Remarks.—This vinegar is prepared by macerating squill in a No. 20 powder with diluted acetic acid for seven days, and then straining through muslin.

The strained liquid is then brought to the boiling-point (in order to coagulate the albumin extracted from the drug), and is filtered while hot.

Since the present official vinegar of squill has been boiled, the official *syrup of squill* prepared from it can be made without boiling of the vinegar, as was directed by the pharmacopœia of 1890.

Dose.—1 Cc. (15 minims).

CHAPTER XVI

OFFICIAL LIQUIDS CONTAINING UNDISSOLVED MATTER

MIXTURES

MIXTURES are aqueous preparations for internal use containing insoluble non-fatty substances. Since they contain insoluble matter, they must be dispensed with a "Shake Well!" label. They differ from emulsions in containing no fat; from liniments, in being used internally.

Up to the revision of 1890 both mixtures and emulsions were grouped under the common head *Mistura*, but at that revision the class *emulsions* was given pharmacopœial recognition.

As mixtures usually contain some organic matter, with water as a vehicle, they are prone to fermentation, and are accordingly unstable. A possible exception to this rule is the compound mixture of glycyrrhiza; but even this popular remedy should not be made in too large a quantity.

TABLE OF OFFICIAL MIXTURES

<i>Process.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparation.</i>
All by simple admixture.	Chalk mixture. Compound mixture of glycyrrhiza. Mixture of rhubarb and soda.	Compound iron mixture.

SPECIAL NOTES ON OFFICIAL MIXTURES

MISTURA CRETÆ. Chalk Mixture.

<i>Recipe</i> .—Compound Chalk Powder, twenty grammes.....	20 Gm.
Cinnamon Water, forty cubic centimeters.....	40 Cc.
Water, a sufficient quantity,	
To make one hundred cubic centimeters..	100 Cc.

Rub the Compound Chalk Powder in a mortar with the Cinnamon Water and about *twenty cubic centimeters* of Water gradually added, to a uniform mixture; transfer this to a graduated vessel, and rinse the mortar with enough Water to make the product measure *one hundred cubic centimeters*. Mix the whole thoroughly. This preparation should be freshly made when wanted.

Remarks.—Chalk mixture is prepared from compound chalk powder (p. 302), water, and cinnamon water. The object of diluting the cinnamon water is because chalk mixture is very often administered to infants, and the amount of oil of cinnamon in the official cinnamon water may be sufficient to cause an irritation of the mouth. Since compound chalk powder contains the ingredients that are easily fermentable,—acacia and sugar,—the mixture should be freshly prepared as needed and dispensed to the patient in small quantities—say, four ounces at a time. It is useful in cases of diarrhea.

Dose.—16 Cc. (4 fluidrachms).

MISTURA FERRI COMPOSITA. Compound Iron Mixture.

This preparation, commonly called *Griffith's mixture*, depends for its activity upon the formation of ferrous carbonate by a chemical reaction between ferrous sulphate and potassium carbonate, hence it can best be considered under the iron salts in Part III.

MISTURA GLYCYRRHIZÆ COMPOSITA. Compound Mixture of Glycyrrhiza.

<i>Recipe.</i> —Pure Extract of Glycyrrhiza, thirty grammes.....	30 Gm.
Syrup, fifty cubic centimeters.....	50 Cc.
Acacia, granulated, thirty grammes.....	30 Gm.
Camphorated Tincture of Opium, one hundred and twenty cubic centimeters.....	120 Cc.
Wine of Antimony, sixty cubic centimeters.....	60 Cc.
Spirit of Nitrous Ether, thirty cubic centimeters....	30 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Rub the Pure Extract of Glycyrrhiza and Acacia in a mortar, with *five hundred cubic centimeters* of Water, until they are dissolved. Transfer the solution to a graduated vessel containing the other ingredients, and rinse the mortar with enough Water to make the product measure *one thousand cubic centimeters*. Mix the whole thoroughly.

Remarks.—This mixture, called *brown mixture*, by reason of its color, is often prescribed in the South under the Latin name "*Mistura Fusca*"—the latter word being Latin for brown. One physician, evidently thinking the recipe was devised by a man named Brown, was known to prescribe it as "*Mistura Browni*."

It might be noted in passing that the brown mixture is composed of an official extract (glycyrrhiza), an official syrup (syrup, U. S. P.), an official gum (acacia), an official wine (antimony), an official spirit (nitrous ether), and an official tincture (paregoric). It is used as a simple remedy in coughs, and for this purpose it is quite efficient.

Dose.—8 Cc. (2 fluidrachms).

MISTURA RHEI ET SODÆ. Mixture of Rhubarb and Soda.

<i>Recipe.</i> —Sodium Bicarbonate, thirty-five grammes.....	35 Gm.
Fluidextract of Rhubarb, fifteen cubic centimeters..	15 Cc.
Fluidextract of Ipecac, three cubic centimeters....	3 Cc.
Glycerin, three hundred and fifty cubic centimeters..	350 Cc.
Spirit of Peppermint, thirty-five cubic centimeters...	35 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Dissolve the Sodium Bicarbonate in about *four hundred cubic centimeters* of Water. Then add the Fluidextracts, the Glycerin, and the Spirit of Peppermint, and, lastly, enough Water to make the product measure *one thousand cubic centimeters*.

Remarks.—This preparation, made from the fluidextracts of rhubarb and ipecac, sodium bicarbonate, and spirit of peppermint, diluted with glycerin and water, is used in some sections as a remedy for flatulence. It does not, however, enjoy universal popularity.

Dose.—4 Cc. (1 fluidrachm).

Doses of Official Mixtures

4 Cc. (1 fluidrachm).....	Mixture of rhubarb and soda.
8 Cc. (2 fluidrachms).....	Mixture of glycyrrhiza compound.
16 Cc. (4 fluidrachms).....	Mixture of chalk and iron compound.

EMULSIONS

Emulsions are aqueous preparations for internal use in which resinous or fatty substances are suspended by means of mucilaginous matter. Like mixtures, they must be shaken before administration. The ideal emulsion is milk, in which the fat (butter) is emulsified by an albuminous substance, casein. In milk the fat-globules are more finely divided than in any artificial emulsion.

Emulsions, like mixtures, are rather unstable, and should be freshly prepared when desired. This question of freshness may be extended to the statement that no emulsion more than a week old should be dispensed. The proprietary emulsions which are marketed quite largely are invariably preserved by adding preservatives, the most innocent of which are glycerin or alcohol.

In making emulsions, druggists have the opportunity of exhibiting the highest degree of pharmaceutic art, and there is no reason why the pharmacist should not make all the emulsions he dispenses, except, of course, those proprietary brands specified by the prescriber. Reserving the discussion of the official emulsions until later, we will first consider the important processes of emulsification.

The emulsion of cod liver oil is the best illustration of an emulsion of a fixed oil. It is manufactured by two methods,—the English method and the Continental method,—and of these, the simplest method of manufacturing is by the Continental method. The secret of the successful manipulation by the Continental method consists in wisely choosing the proportions of gum water and oil employed, these proportions being: oil, 4 parts; water, 2 parts; and gum, 1 part; or, to express it in other words, use twice as much water as gum, and twice as much oil as water. The manipulation is fully explained on p. 1113, and consists of triturating the gum with the oil, and then adding the water all at once, and stirring rapidly until the emulsion results. The emulsion nucleus, as it is called, can be easily diluted with water, syrup, and flavors to the required quantity, it being the general custom in this country to dispense the emulsion of cod liver oil containing 50 per cent. of oil. The objection to the 50 per cent. acacia emulsion is that the resulting product is quite thick and difficult to pour out from a bottle; and, moreover, the use of a large

quantity of gum arabic required makes the emulsion rather expensive. For this reason, other gums have been used besides gum arabic, such as tragacanth, Irish moss, gelatin, and dextrin, and recipes for such emulsions can be found in the National Formulary.

Powdered gelatin has also been used in the manufacture of emulsions, and when a combination of powdered gelatin and tragacanth is used, the emulsion can be made in a bottle without the use of a mortar. Powders for the manufacture of these so-called flask emulsions were exploited a few years since, and the best known of them, when examined by the author, was found to consist of saponin, saccharin, and a small quantity of a gummy substance. Neither saponin nor saccharin should be used indiscriminately; it is hardly necessary to state that the pharmacist should be chary in employing such substances in emulsions. As for flavoring, various aromatic volatile oils are employed, the present pharmacopœia directing such use of either oil of gaultheria or oil of bitter almond.

If an emulsion of castor oil is required, it is made like emulsion of cod liver oil, and the recipe for the same can be found in the National Formulary.

In making emulsions of fixed oils according to the English method, the process consists of triturating gum with water, thus making a mucilage, and adding a small quantity of oil, then adding a little water, and then a little oil, and so on, adding the oil and water alternately until the emulsions are completed. This method of manufacturing the emulsions is slow and uncertain, and cannot be compared in simplicity with the preparation of emulsions by the Continental method. It has, however, the advantage that the comparatively large amount of oil can be emulsified by the addition of a small quantity of gum; and when a physician, therefore, prescribes an emulsion containing less gum than is required by the method of manufacturing according to the Continental method, the English method of emulsification is indicated.

Very frequently the manufacture of emulsion results in a failure, and the product, instead of being a creamy-white product, is an ugly, greasy mixture. This failure in making emulsions is what is called "splitting" an emulsion. Such a split emulsion can be redeemed by adding it gradually to a concentrated perfect emulsion, following the dictum that "the best emulsifier is a perfect emulsion."

Emulsions of volatile oils can be prepared either with gum arabic or tragacanth, although the latter, in the hands of the author, gives more satisfactory results. The manufacture of a typical emulsion of a volatile oil is found in the process given for making emulsion of turpentine (U. S. P.), in which recipe acacia is used as the emulsifier. The recipe can be used for any other volatile oil by substituting such volatile oil for the oil of turpentine in the official recipe.

In all emulsions that we have thus far considered the emulsifying agent is a gummy substance. Such emulsions have decided limitations, however, the gum being thrown out of the combination by the addition of alcohol, glycerin, or tincture of iron in anything more than in small quantities. For this reason the best emulsifying agent is yolk of egg, and the recipe for emulsion of cod liver oil made with a yolk of egg as a base is found in the National Formulary. Another

egg emulsion of cod liver oil is one devised by the author, which is found in Part VI.

Extract of malt is also a valuable emulsifying agent, and the recipe for an emulsion made in this way is as follows: Extract of malt, 1 part; triturate with cod liver oil, 4 parts; then add syrup of wild cherry, 2 parts, and sherry wine, 1 part.

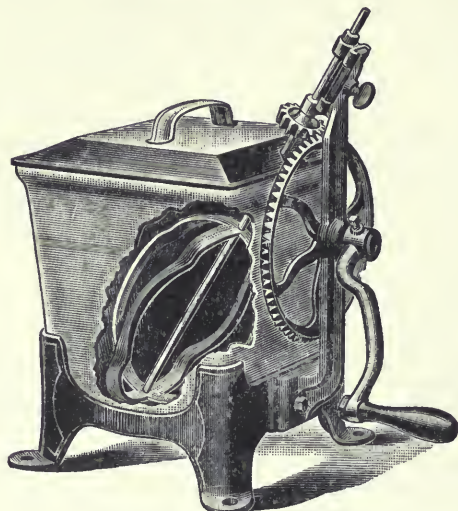


Fig. 166.—Emulsion machine.

Casein has also been suggested as an emulsifying agent, it being the emulsifier in the ideal emulsion, milk. In the writer's hands, however, the commercial casein has never yielded satisfactory results, nor has he been any more fortunate when using condensed milk, which has been suggested as an emulsifying agent. In working up large quantities of emulsions, a power machine is frequently advisable. Such a machine, operating by hand, is shown in Fig. 166.

TABLE OF OFFICIAL EMULSIONS

(ALL GALENIC)

Gum-resin emulsion.....	Emulsion of asafetida.
Fixed oil emulsions.....	Emulsion of almond.
	Emulsion of chloroform.
	Emulsion of cod liver oil.
	Emulsion of cod liver oil with hypophosphites.
Volatile oil emulsion.....	Emulsion of oil of turpentine.

SPECIAL NOTES ON OFFICIAL EMULSIONS

EMULSUM AMYGDALÆ. Emulsion of Almond.

<i>Recipe.</i> —Sweet Almond, sixty grammes.....	60 Gm.
Acacia, in fine powder, ten grammes.....	10 Gm.
Sugar, thirty grammes.....	30 Gm.
Water, a sufficient quantity,	

To make one thousand cubic centimeters.. 1000 Cc.

Having blanched the Almond, add the Acacia and Sugar, and beat them in a mortar until they are thoroughly mixed. Then rub the mass with *nine hundred*

cubic centimeters of Water, at first very gradually added, until a uniform mixture results. Strain this into a graduated vessel, and wash the mortar and strainer with enough Water to make the product measure *one thousand cubic centimeters*. Mix the whole thoroughly. This Emulsion should be freshly prepared when required.

Remarks.—While this is grouped in the foregoing table as a fixed oil emulsion, we make it from sweet almond (seeds), emulsifying the oil existing therein with gum arabic. The proteids of the almond seed also assist in the emulsification. Note that the almonds must be blanched (p. 735).

The emulsion of almonds are also known as *milk of almonds*, and is used as a pleasant vehicle in medicine, although its use in this country is rather limited.

Dose.—120 Cc. (4 fluidounces).

EMULSUM ASAFETIDÆ. Emulsion of Asafetida.

Recipe.—Asafetida, in selected tears, *forty grammes*..... 40 Gm.

Water, *a sufficient quantity*,

To make *one thousand cubic centimeters*.. 1000 Cc.

Rub the Asafetida in a mortar, with *nine hundred cubic centimeters* of Water, at first very gradually added, until a uniform emulsion results. Then strain the mixture into a graduated vessel, and rinse the mortar and strainer with enough Water to make the product measure *one thousand cubic centimeters*. Mix the whole thoroughly.

Remarks.—Asafetida, as explained on p. 856, is a gum-resin; it consists of a water-insoluble resin and a water-soluble gum. If we rub asafetida with water, the gum, on dissolving, will emulsify the resin, and a milky mixture, familiarly called *milk of asafetida*, results.

In making this emulsion the whole drug asafetida is placed in a mortar and triturated with water until the emulsion results. Under no circumstances should the powdered drug be used, since the preparation owes its activity to volatile oils, and in getting the drug brittle enough to powder it is necessary to dry it by heat, and this results in driving off an appreciable quantity of the oils. In order to make a good emulsion the best quality of asafetida (tears) should be employed.

The exceedingly unpleasant and persistent odor of asafetida has led to methods of preparing the emulsion in some way other than of triturating the drug in a mortar, for removing the odor from the mortar after making the emulsion is a matter of difficulty. Moreover, since emulsion of asafetida should be freshly prepared, it means that each time a small quantity of the emulsion is called for it is necessary to use the mortar, with the result of keeping it in a continuous state of malodorousness. One method in vogue among certain druggists for making this emulsion—by diluting the tincture of asafetida with water—is nothing short of criminal, and under no circumstances should it be followed.

A less worthy method, but one which gives fairly good results, is by preparing a glycerite of asafetida by gently warming the gum-resin with glycerin, and thus obtaining a preparation eight times stronger than the emulsion. In preparing the emulsion from this glycerite one drachm of the glycerite is taken and diluted with seven drachms of water. The chief objection to the manufacture of gly-

cerite of asafetida is the uncertainty that the drachm of the glycerite will contain the full active amount of asafetida required for an ounce of the full-strength finished product.

After all, the only safe way is the official method, and as an illustration of financial advantage of being "faithful in small things" may be cited the case of a pharmacist who built up a large prescription business from comparatively small beginnings by calling the attention of physicians in his city to the fact that he made this emulsion, as well as other pharmacopœial staples, "just right."

The disagreeable odor clinging to the utensils used in preparing this emulsion may be combated in two ways.

Best of all is it to have a special mortar for asafetida and other evil-smelling drugs. If that is not feasible, the mortar can be freed from odor by igniting a small amount of alcohol (say one to two fluidrachms) in the mortar, and triturating the burning liquid with pestle until it is burned out. It is needless to say that the pestle handle must be fitted with plaster-of-Paris cement and not with shellac (p. 114), for the latter cement would be softened by the heat of burning alcohol and the handle become loose. Moreover, this method must be cautiously employed, as the sudden heat sometimes cracks the mortar. The same method is of value in removing the odor of iodoform from a mortar.

Dose.—16 Cc. (4 fluidrachms).

EMULSUM AMMONIACI (U. S. P. 1890). Emulsion of Ammoniac.

This official of the pharmacopœia of 1890, dropped in the last revision, is a 4 per cent. emulsion of the gum-resin, ammoniac, made by triturating the drug, in tears, with water.

EMULSUM CHLOROFORMI. Emulsion of Chloroform.

<i>Recipe.</i> —Chloroform, <i>forty cubic centimeters</i>	40 Cc.
Expressed Oil of Almond, <i>sixty cubic centimeters</i> ...	60 Cc.
Tragacanth, in very fine powder, <i>ten grammes</i>	10 Gm.
Water, <i>a sufficient quantity</i> ,	

To make *one thousand cubic centimeters*.. 1000 Cc.

Introduce the Tragacanth into a perfectly dry bottle of sufficient capacity, add the Chloroform, and shake the bottle thoroughly, so that every part of the surface may become wetted. Then add about *two hundred and fifty cubic centimeters* of Water, and incorporate it by vigorous shaking. Next add the Expressed Oil of Almond, in several portions, shaking after each addition, and when the Oil has been thoroughly emulsified, add enough Water, in divided portions, shaking after each addition, to make the product measure *one thousand cubic centimeters*.

Remarks.—In making this emulsion, tragacanth is the emulsifying agent, the chloroform being assisted to minute subdivision by means of expressed oil of almond, which also aids in making the emulsion permanent. Note that this is one of the so-called "flask emulsions," being made by shaking the ingredients in a bottle, saving the soiling of a mortar. Another official flask emulsion is emulsion of oil of turpentine, and it might be added that all emulsions of volatile oils can be so made. Emulsions of fixed oils and of gum-resins, on the other hand, should always be made in a mortar.

Dose.—8 Cc. (2 fluidrachms)..

EMULSUM OLEI MORRHUÆ. Emulsion of Cod Liver Oil.

<i>Recipe</i> .—Cod Liver Oil, <i>five hundred cubic centimeters</i>	500 Cc.
Acacia, in fine powder, <i>one hundred and twenty-five grammes</i>	125 Gm.
Syrup, <i>one hundred cubic centimeters</i>	100 Cc.
Oil of Gaultheria, <i>four cubic centimeters</i>	4 Cc.
Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Rub the Acacia with the Cod Liver Oil in a dry mortar until uniformly mixed, then add at once *two hundred and fifty cubic centimeters* of Water and triturate lightly and rapidly until a thick homogeneous emulsion is produced; to this add the Oil of Gaultheria and the Syrup, with enough Water to make the product measure *one thousand cubic centimeters*, and mix thoroughly.

Note.—The Oil of Gaultheria may be replaced, if desired, by a suitable quantity of Oil of Bitter Almond or other suitable flavoring.

Remarks.—Every detail of manipulation of this valuable preparation will be found in Part VI.

Dose.—8 Cc. (2 fluidrachms).

EMULSUM OLEI MORRHUÆ CUM HYPOPHOSPHITIBUS. Emulsion of Cod Liver Oil with Hypophosphites.

Recipe.—Like recipe for emulsion of cod liver oil, except that 10 Gm. calcium hypophosphite, 5 Gm. sodium hypophosphite, and 5 Gm. potassium hypophosphite are dissolved in 100 Cc. water and mixed with syrup, and then with the emulsion nucleus produced by triturating 500 Cc. oil, 250 Cc. water, and 125 Gm. acacia.

Remarks.—This is the popular “cod liver oil with hypophosphites” so largely prescribed for wasting diseases.

Dose.—8 Cc. (2 fluidrachms).

EMULSUM OLEI TEREBINTHINÆ. Emulsion of Oil of Turpentine.

<i>Recipe</i> .—Rectified Oil of Turpentine, <i>fifteen cubic centimeters</i>	15 Cc.
Expressed Oil of Almond, <i>five cubic centimeters</i>	5 Cc.
Syrup, <i>twenty-five cubic centimeters</i>	25 Cc.
Acacia, in fine powder, <i>fifteen grammes</i>	15 Gm.
Water, <i>a sufficient quantity</i> ,	
To make <i>one hundred cubic centimeters</i> ..	100 Cc.

Introduce the Acacia into a perfectly dry bottle of sufficient capacity, add the Rectified Oil of Turpentine and the Expressed Oil of Almond, and shake the bottle thoroughly. Then add about *thirty cubic centimeters* of Water and incorporate it by vigorous shaking. When the Oil has been completely emulsified, add first the Syrup, in several portions, shaking after each addition, and then enough Water, in divided portions, shaking after each addition, to make the product measure *one hundred cubic centimeters*.

Remarks.—Every detail of manipulation of this recipe will be given in Part VI. Note that it contains 15 per cent. oil of turpentine, and the rectified oil at that. Note that a small amount of expressed oil of almond is added to facilitate emulsification.

Dose.—4 Cc. (1 fluidrachm).

Doses of Official Emulsions

4 Cc. (1 fluidrachm).....	Emulsion of oil of turpentine.
8 Cc. (2 fluidrachms).....	Emulsions of chloroform, cod liver oil, and cod liver oil with hypophosphites.
16 Cc. (4 fluidrachms).....	Emulsion of asafetida.
120 Cc. (4 fluidounces).....	Emulsion of almond.

LINIMENTS

Liniments are liquid preparations for external use, applied by friction. Not all official liniments contain insoluble matter, for, as shown in the table of preparations (p. 166), some are clear alcoholic solutions; in fact, but two of the official liniments—ammonia and lime liniments—are opaque. But since some of the rest are alcoholic solutions,—such as soap liniment,—and some are clear oleaginous solutions,—such as camphor liniment,—and one is a more or less transparent semisolid mass,—turpentine liniment,—there seems no definite basis of pharmaceutic grouping of liniments, hence they were left under “liquids containing insoluble matter,” to be the last class of pharmaceutic liquids to be discussed.

TABLE OF OFFICIAL LINIMENTS

<i>Consistence.</i>	<i>Basis.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparations.</i>
Clear liquids.	Alcoholic.	Alcohol.	Soap liniment.
		Soap liniment.	Soft soap liniment.
		Fluidextract.	Chloroform liniment.
Opaque liquids.	Oleaginous.	Cotton seed oil.	Belladonna liniment.
		Cotton seed oil.	
Semisolid.	Oleaginous.	Linseed oil.	Ammonia liniment.
		Oil of turpentine.	Lime liniment.
		Turpentine liniment.	

SPECIAL NOTES ON OFFICIAL LINIMENTS

LINIMENTUM AMMONIÆ. Ammonia Liniment.

Recipe.—This preparation, commonly called *hartshorn liniment*, or *volatile liniment*, is a variety of soap, and will, therefore, be considered among the soaps (Part IV.).

LINIMENTUM BELLADONNÆ. Liniment of Belladonna.

Recipe.—Camphor, *fifty grammes*..... 50 Gm.
Fluidextract of Belladonna Root, a sufficient
quantity,

To make one thousand cubic centimeters... 1000 Cc.

Dissolve the Camphor in about *eight hundred cubic centimeters* of the Fluidextract, and then add enough of the latter to make the product measure *one thousand cubic centimeters*. Mix thoroughly.

Remarks.—This consists of the fluidextract of belladonna in which 5 per cent. of camphor is dissolved.

LINIMENTUM CALCIS. Lime Liniment.

This liniment (Carron oil) is a variety of lime soap, and will, therefore, be discussed in Part IV.

LINIMENTUM CAMPHORÆ. Camphor Liniment.

<i>Recipe.</i> —Camphor, in coarse powder, two hundred grammes...	200 Gm.
Cotton Seed Oil, eight hundred grammes.....	800 Gm.
To make one thousand grammes..	1000 Gm.

Introduce the Camphor and the Cotton Seed Oil into a suitable flask, and apply a gentle heat, by means of a water-bath, loosely stoppering the flask during the operation. Agitate the flask occasionally until the Camphor is dissolved.

Remarks.—This liniment, which is commonly called *camphorated oil*, is made by dissolving 200 Gm. of camphor in 800 Gm. of cotton seed oil. In preparing this liniment the pharmacist should be very careful in choosing a winter-strained cotton seed oil, that is, an oil that has been filtered during the cold weather, and, therefore, is freed from dissolved solid matter. If the so-called summer-strained oil is used in making such a liniment, on the first subjection to cold the liniment will become cloudy, and will scarcely reflect credit upon the pharmacist from whom it was bought.

LINIMENTUM CHLOROFORMI. Chloroform Liniment.

<i>Recipe.</i> —Chloroform, three hundred cubic centimeters.....	300 Cc.
Soap Liniment, seven hundred cubic centimeters....	700 Cc.
To make one thousand cubic centimeters..	1000 Cc.

Mix them by agitation.

Remarks.—This liniment is made by mixing 700 Cc. soap liniment and 300 Cc. chloroform, and is one of the most valuable of penetrating liniments.

LINIMENTUM SAPONIS. Soap Liniment.

<i>Recipe.</i> —Soap, dried and granulated, sixty grammes.....	60 Gm.
Camphor, in small pieces, forty-five grammes.....	45 Gm.
Oil of Rosemary, ten cubic centimeters.....	10 Cc.
Alcohol, seven hundred and twenty-five cubic centimeters.....	725 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Add the Soap to two hundred cubic centimeters of boiling Water, heat the mixture on a water-bath until a clear gelatinous mass results. Mix this, while yet warm, with five hundred cubic centimeters of Alcohol, and stir it until solution is effected. Dissolve the Camphor and Oil of Rosemary in two hundred and twenty-five cubic centimeters of the Alcohol by agitation in a bottle; add this solution to the warm Soap mixture; mix thoroughly, and, if necessary, add enough Water to make the product measure one thousand cubic centimeters. Set it aside in a cool place for twenty-four hours, then filter.

Remarks.—Soap liniment is the base of chloroform liniment, and is used quite largely for the extemporaneous preparation of a number of liniments. Thus it is used in preparing a liniment of quinine, which, when applied by friction to children, produces the physiologic effects of quinine without it being taken in by the mouth.

Soap liniment is sometimes called *liquid opodeldoc*, the original being a preparation similar to the official soap liniment, but containing enough soap to render it solid at ordinary temperatures. When the solid opodeldoc is warmed gently, it liquefies. A recipe for solid opodeldoc is found in the National Formulary.

LINIMENTUM SAPONIS MOLLIS. Liniment of Soft Soap.

<i>Recipe.</i> —Soft Soap, <i>six hundred and fifty grammes</i>	650 Gm.
Oil of Lavender Flowers, <i>twenty cubic centimeters</i> ..	20 Cc.
Alcohol, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Mix the Oil of Lavender Flowers with *three hundred cubic centimeters* of Alcohol, dissolve in this the Soft Soap by stirring or agitation, and set the solution aside for twenty-four hours. Then filter it through paper, adding sufficient Alcohol to make the product measure *one thousand cubic centimeters*.

Remarks.—This was called *tincture of green soap* in the pharmacopœia of 1880, and is prepared by dissolving soft soap (65 per cent.) in alcohol and flavoring with oil of lavender. It is used almost exclusively as an application to the scalp, and is, therefore, the basis of a number of hair-tonics.

LINIMENTUM TEREBINTHINÆ. Turpentine Liniment.

<i>Recipe.</i> —Rosin Cerate, <i>six hundred and fifty grammes</i>	650 Gm.
Oil of Turpentine, <i>three hundred and fifty grammes</i> ..	350 Gm.
To make <i>one thousand grammes</i> ..	1000 Gm.

Dissolve the Rosin Cerate, previously melted in a dish on a water-bath, in the Oil of Turpentine, and mix them thoroughly.

Remarks.—This is the only official liniment made with oil of turpentine as a base. It consists of resin cerate thinned with the volatile oil, and is a semisolid mass that should be dispensed in an ointment jar.

Doses of Liniments.—As all liniments are used externally, no doses are given.

CHAPTER XVII

EXTRACTS, ABSTRACTS, RESINS

THESE three classes of preparations include the only solid pharmaceuticals prepared by percolation.

EXTRACTS

Extracts are solid or semisolid preparations of the soluble and active principles of drugs prepared by percolation of the drug with the appropriate menstruum and evaporation of the percolate.

The menstruum employed varies as much as that directed for fluidextracts, being alcoholic, hydro-alcoholic, aqueous, alcoholic and alkaline, or alcoholic and acid. Besides extracts so prepared, nine of the extracts of the present pharmacopœia are made by evaporating the fluidextract.

A special form of extracts, called *inspissated juices*, are popular in England, but have no representative in the present pharmacopœia of the United States. They were, however, represented in the pharmacopœia of 1880 by the then official extract of taraxacum. The present official extract of taraxacum is not an inspissated juice, but is prepared by percolation of the drug with hydro-alcoholic menstruum. Extract of taraxacum (U. S. P. 1890) was scarcely a fair type of the inspissated juices, the most prominent of these being the

dried juices from the leaves of plants, and, therefore, containing chlorophyl. Such inspissated juices are prepared by bruising fresh leaves or herbs in a stone mortar, pouring off the greenish liquid, and allowing it to stand over night. The liquid is then strained through calico, thereby separating the chlorophyl; the strained liquid is heated to 200° F., which coagulates the albumin present, and this is removed by straining. The clear liquid is then evaporated to a thin extract, to which is added the chlorophyl, and the whole mixture is evaporated until it is of pilular consistence. Were the plant-juices evaporated to extract directly on separating from the plant, the delicate chlorophyl would be quickly decomposed and a brown extract would result.

By first removing the chlorophyl and evaporating the mixture to a thin extract, the chlorophyl is preserved. When added just as the extract is reaching pilular consistence, it has the effect of giving to it an agreeable green color.

Such inspissated juices, as of aconite, belladonna, and hyoscyamus, are very popular in England, but are scarcely used in this country, and it should be borne in mind that these English "extracts" are much weaker than are the official American extracts.

TABLE OF OFFICIAL EXTRACTS
(ALL GALENIC)

<i>Process.</i>	<i>Preparation.</i>	<i>Consistence.</i>
Percolation with evaporation:		
Menstruum,		
Alcohol, U. S. P.	{ Extract of Indian cannabis.	Soft.
Alcohol, 2000 Cc. }	{ Extract of physostigma.	Powdered.
Water, 1000 Cc. }	 Extract of belladonna leaves.	Soft.
Diluted Alcohol:	Extract of colocynth.	Powdered.
Alcohol, 1000 Cc. }	 Extract of ergot.	Soft.
Water, 400 Cc. }	{ Extract of cascara sagrada.	Powdered.
Alcohol, 125 Cc. }	{ Extract of taraxacum.	Soft.
Water, 875 Cc. }	 Extract of colchicum corm.	Soft.
Acetic acid, 350 Cc. }	 Extract of nux vomica.	Powdered.
Water, 1500 Cc. }	 Extract of glycyrrhiza, pure.	Soft.
Acetic acid, 500 Cc. }		
Water, 1300 Cc. }		
Ammonia water, 150 Cc. }		
Water, 3000 Cc. }		
Water	{ Extract of gentian.	Soft.
	{ Extract of krameria.	Powdered.
	{ Extract of quassia.	Powdered.
Evaporation of the fluidextract:	Extract of cimicifuga.	Powdered.
	Extract of digitalis.	Soft.
	Extract of euonymus.	Powdered.
	Extract of hyoscyamus.	Soft.
	Extract of leptandra.	Powdered.
	Extract of rhubarb.	Soft.
	Extract of scopola.	"
	Extract of stramonium.	"
	Extract of sumbul.	"
Evaporation of infusion or decoction:	Extract of aloes.	Powdered.
	Extract of hematoxylon.	"
	Extract of malt.	Soft.
	Extract of opium.	Powdered.
A commercial product:	Extract of glycyrrhiza.	
A compound extract:	Extract of colocynth, compound.	

Of these 28 extracts, 13 are powdered, while 15 are soft, the last class including those of pilular consistence; one thin as honey (extract of malt); and one almost brittle (extract of glycyrrhiza).

Two are *alcoholic*—that is, made with alcohol, U. S. P., as menstruum (3, if extract of cimicifuga, made by evaporating the fluidextract, is included); 5 are hydro-alcoholic (13, if those made by evaporating the fluidextract are included); 7 are *aqueous*, that is, made by evaporating the watery percolate, infusion, or decoction; 2 are *acetic*; that is, made by extracting drug with a diluted acetic acid; and one is made by extracting drug with water containing ammonia.

Taking up the several methods of preparing extracts outlined in the table just given, a few words of general explanation of each process may be in place at this time, leaving, of course, minutiae for consideration under Special Notes.

In the process of *percolation with evaporation*, when the menstruum is hydro-alcoholic, the drug is percolated with the official menstruum, the first portion of the percolate (usually representing in cubic centimeters 90 per cent. of the weight in grammes of the drug) is put aside as a reserve portion. The remainder of the percolate is evaporated at a low temperature to a very small bulk. With this the reserve portion is mixed, and the mass evaporated to pilular consistence.

The manufacture of extracts by this process is one portion of pharmaceutical manufacturing which cannot be done as well by the retail pharmacist as by the large manufacturer. In order to successfully evaporate the extract at a low temperature—say, 50° C.—it is necessary that a vacuum apparatus be employed, and the cost of such an apparatus precludes its use by the retail pharmacist.

In other cases where alcoholic menstruum is employed, none of the percolate is set aside as reserved portion, but the entire percolate is directly evaporated. This is the case where alcohol, U. S. P., is the menstruum, or where the constituents of the drug are not easily injured by heat. Likewise, if the menstruum is water or a diluted acetic acid, there is no reserve portion, but the entire percolate is evaporated.

The process of *evaporation of the fluidextract*, which was recognized in the pharmacopœia of 1890 in only one case (extract of ergot), but which constitutes the official recipe in nine extracts of the present pharmacopœia, is really a modification of the process of percolation with evaporation; since the fluidextracts, themselves prepared by percolation and possibly partial evaporation, are in the recipe evaporated. One point regarding this process worthy of note is that six of the extracts so prepared are directed to be of pilular consistence, while three are to be powdered extracts, and that these powdered extracts are prepared by incorporation of enough powdered glycyrrhiza to make the extract dry and pulverulent. In passing, it might be stated that the pharmacopœia (Introduction, p. lii) permits use of exhausted marc of the drug instead of powdered glycyrrhiza.

The process of *evaporation of infusion or decoction* simply consists of extracting the drug with water—either by infusion or decoction—and evaporation of the watery solution either to pilular consistence or by finishing of the product as a powdered extract. As mentioned above, extracts are made pulverulent by addition of some inert pow-

der. Glycyrrhiza is frequently the diluent, but in many cases, as in extract of opium, sugar of milk is employed. Usually enough diluent is added to make the product of definite strength. Of this, mention will be made when we consider strength of extracts.

In many respects soft extracts are the least satisfactory of all the official preparations. Thus the phrase, "evaporate to pilular consistence," is rather vague, and the strength of the finished product is apt to be uncertain; not only when first made, but also because, on standing, the strength is apt to change by loss of some of the water the extract contains, the extract becoming hard and dry.

To prevent this, the pharmacopœia permits the addition, when necessary, of 10 per cent. of glycerin to the extract which is to be kept of a pilular consistence.

A second factor in the uncertainty of the pilular extracts is the amount of extractive which is contained therein. By the word "extractive" is meant the soluble and inert constituents naturally residing in drugs, grouped, in most text-books of materia medica, as gums, starches, and sugars. These constituents are practically useless from a therapeutic standpoint, and could they be eliminated, the extract would be much stronger. The question of the quantity of the extractive found in the extracts depends entirely on the menstruum employed, one containing a large amount of water yielding a larger amount of extract than those of a strongly alcoholic nature.

The question of the appropriate menstruum for extracting the full strength of the drug without excess of inert matter has been carefully studied by the Committee on Revision of the Pharmacopœia, and the menstruum directed by that standard is the one yielding the most satisfactory results. This menstruum should, therefore, be strictly followed. Yet there have been known unscrupulous manufacturers who deliberately deviate from the pharmacopœial menstruum, adding more water than directed by that standard, in order that large amounts of extract may be obtained. In such cases, of course, an increased yield means a diminution in the active strength of the extract, and it behooves the pharmacist to purchase extracts only from those manufacturers who are known to be reliable.

Among the many constituents which are included in the word "extractive" are the mineral salts, found naturally existing in drugs, such as potassium chloride and sulphate. From many moist extracts on standing the salts crystallize, and such extracts, when rubbed up in making an ointment, are frequently found to be gritty. Whether the grittiness of such extracts is due to crystals or to the accidental mixture of fragments of glass in the container, can be proved by adding a few drops of water and rubbing with an ointment spatula. Should the grittiness dissolve, it shows that it was due to these natural salts; whereas if the grittiness remains after trituration, fragments of glass are indicated.

A third disadvantage in the use of soft extracts is purely pharmaceutical, and that is the inconvenience in handling a semisolid mass. Extracts, especially those of potent drugs, are frequently prescribed in small quantities, say from $\frac{1}{4}$ to $\frac{1}{2}$ grain. To weigh out the soft extracts it is necessary that the $\frac{1}{4}$ to $\frac{1}{2}$ grain be weighed on a piece of paper, and by the time the quantity is scraped off the paper and

off the spatula, there is frequently very little of the extract transferred to the medicine. It is hardly necessary to say that in weighing such extracts the quantity should be transferred to the paper from the container by means of a perfectly clean spatula, and the spatula cleaned before transferring the weighed portion of the extract into the mortar or other mixture with which it is to be combined. If the surface of paper on which the extract is to be weighed is moistened before "taring," the extract will not stick so closely. Still better is it to use oiled paper or parchment paper.

A fourth inconvenience connected with soft extracts is the uncertainty of the dosage of the extract, due to the uncertainty of the strength of this extract. The variability of the extract-strength is of a twofold character: first, different drugs yield vastly differing quantities of extracts. Thus, gum opium yields about half its weight of soft extract; extract of belladonna and extract of hyoscyamus yield about one-fifth their weight of extracts, while extract of nux vomica is about ten times stronger than the drug from which it is obtained.

Hence it is seen that different drugs yield vastly different quantities of extract; not only this, but, worst of all, different batches of the same drug yield different quantities of extract; a very important factor in quality of drugs being the soil on which they have been raised, and particularly the season in which they are collected.

Therefore at their best soft extracts are inconvenient and unreliable, and the revision committee did well in directing that so many of the extracts of the present pharmacopœia be powdered and of definite strength, and that the extracts from potent drugs be assayed.

Strength of Extracts.—As just mentioned, many of the extracts of the present pharmacopœia are directed to be of definite strength. A list of these standardized extracts is here given:

OFFICIAL ASSAYED SOFT EXTRACTS

<i>Name.</i>	<i>Assayed standard.</i>	<i>Percentage of extract to drug.</i>	<i>Diluent used.</i>
Extract of belladonna leaves.	1.4 per cent. mydriatic alkaloids.	400	Sugar of milk.
Extract of colchicum corm.	1.4 per cent. colchicine.	400	" " "
Extract of hyoscyamus.	0.3 per cent. mydriatic alkaloids.	about 400	" " "
Extract of scopola.	2.0 per cent. mydriatic alkaloids.	400	" " "
Extract of stramonium.	1.0 per cent. mydriatic alkaloids.	285	" " "

OFFICIAL ASSAYED POWDERED EXTRACTS

<i>Name.</i>	<i>Assayed standard.</i>	<i>Percentage of extract to drug.</i>	<i>Diluent used.</i>
Extract of nux vomica.	5 per cent. strychnine.	400	Sugar of milk.
Extract of opium.	20 per cent. morphine.	about 200 of moist or 160 of powdered.	" " "
Extract of physostigma.	2 per cent. ether-soluble alkaloids.	1333	Powdered glycyrrhiza.

OFFICIAL STANDARDIZED POWDERED EXTRACTS

<i>Name.</i>	<i>Standard.</i>	<i>Percentage of extract to drug.</i>	<i>Diluent used.</i>
Extract of cascara sagrada.	250 Gm. from 1000 Gm. drug.	400	Powdered glycyrrhiza.
Extract of cimicifuga.	25 Gm. from 100 Cc. fluidextract.	400	Powdered glycyrrhiza.
Extract of euonymus.	25 Gm. from 100 Cc. fluidextract.	400	Powdered glycyrrhiza.
Extract of leptandra.	25 Gm. from 100 Cc. fluidextract.	400	Powdered glycyrrhiza.
Extract of quassia.	100 Gm. from 1000 Gm. drug.	1000	Sugar of milk.

OFFICIAL STANDARDIZED SOFT EXTRACTS

<i>Name.</i>	<i>Standard.</i>	<i>Percentage of extract to drug.</i>	<i>Diluent used.</i>
Extract of ergot.	125 Gm. from 1000 Gm. drug.	800	Glycerin.

The preservation of extracts should be a matter of more concern than is usually given the subject in the retail pharmacy. Soft extracts should be kept covered sufficiently tight to prevent undue evaporation; since this causes them to become hard, dry lumps. A capital way of keeping extracts is by placing the original containers in which they are obtained from the manufacturer in special extract jars, and thus each extract is protected from the dust.

Powdered extracts—especially those diluted with sugar of milk—should be kept in well-stoppered bottles in a dry place. In contact with moist air they absorb moisture and become sticky masses.

In such cases desiccation and subsequent pulverization will restore the extract to its original form.

Commercial extracts—usually prepared by evaporation in a copper vacuum pan—are frequently contaminated with *copper*. This can be detected by sticking a bright steel spatula into the jar of suspected extract and allowing it to remain over night. If copper is present, a coating of the metal will be found on the spatula in the morning.

SPECIAL NOTES ON OFFICIAL EXTRACTS

EXTRACTUM ALOES. Extract of Aloes.

Recipe.—Aloes, one hundred grammes..... 100 Gm.
Boiling Water, one thousand cubic centimeters..... 1000 Cc.

Mix the Aloes with the Boiling Water in a suitable vessel, stirring constantly until the particles of Aloes are thoroughly disintegrated, and allow the mixture to stand for twelve hours; then pour off the clear liquid, strain the residue, and evaporate the mixed liquids to dryness with the aid of a water-bath or steam-bath.

Remarks.—This extract is prepared by evaporating an aqueous infusion of aloes to dryness.

Dose.—125 milligrammes (2 grains).

EXTRACTUM BELLADONNÆ FOLIORUM. Extract of Belladonna Leaves.

Recipe.—Belladonna Leaves, in No. 60 powder, one thousand grammes..... 1000 Gm.
Alcohol,
Water, each, a sufficient quantity.

Mix *two thousand cubic centimeters* of Alcohol with *one thousand cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding menstruum, using the same proportions of Alcohol and Water as before, until *three thousand cubic centimeters* of tincture are obtained, or the *Belladonna Leaves* are exhausted. Reserve the first *nine hundred cubic centimeters* of the percolate, evaporate the remainder, at a temperature not exceeding 50° C. (122° F.), to *one hundred cubic centimeters*, mix the residue with the reserved portion, and evaporate at or below the above-mentioned temperature to a pilular consistence.

When assayed, Extract of *Belladonna Leaves* should contain 1.4 per cent. of mydriatic alkaloids. If the Extract should be found by the assay to contain more than this percentage, sufficient powdered sugar of milk should be added to reduce it to the standard of 1.4 per cent.

Remarks.—The above recipe is typical of those fluidextracts prepared by percolation, of the drug with hydro-alcoholic menstruum, reserving a certain portion of the percolate, evaporation of the rest of the percolate, eventually adding the reserved portion, and finally assaying the finished product, using sugar of milk as a diluent if necessary. For Assay, see Chapter LV.

This extract was called by the pharmacopœia of 1890 *alcoholic extract of belladonna leaves*, to differentiate it from the *English extract of belladonna leaves*, which, as mentioned above, is an inspissated juice, the strength of which is much weaker than the official extract.

This extract is a constituent of the official *ointment of belladonna*, of *compound laxative pills*, and of *pills of podophyllum, belladonna, and capsicum*.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

EXTRACTUM CANNABIS INDICÆ. Extract of Indian Cannabis.

Recipe.—Indian Cannabis, in No. 20 powder, *one thousand grammes* 1000 Gm.
Alcohol, a sufficient quantity.

Moisten the powder with *three hundred cubic centimeters* of Alcohol, and pack it firmly in a cylindrical percolator; then add enough Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding Alcohol, until the Indian Cannabis is exhausted. Distil off the Alcohol from the percolate by means of a water-bath, and evaporate the residue, in a porcelain dish, on a water-bath, to a pilular consistence.

Remarks.—This recipe differs from that for extract of *belladonna leaves* in omitting reserve portion of percolate and in directing distillation of alcohol from the percolate and evaporation on water-bath to pilular consistence, without regard to definite strength.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

EXTRACTUM CIMICIFUGÆ. Extract of Cimicifuga.

Recipe.—Fluidextract of Cimicifuga, *one hundred cubic centimeters* 100 Cc.
Glycyrrhiza (peeled, Russian), in No. 80 powder,
a sufficient quantity,
To make twenty-five grammes. . . 25 Gm.

Evaporate the Fluidextract of *Cimicifuga* in a porcelain dish, by means of a water-bath, at a temperature not exceeding 70° C. (158° F.), with constant stirring, to complete dryness. Reduce the product to a fine powder and add enough powdered Glycyrrhiza to make the finished Extract weigh *twenty-five grammes*. Mix thoroughly.

Remarks.—This recipe is typical of those official fluidextracts prepared by evaporating the fluidextract and adding enough powdered glycyrrhiza to make finished product pulverulent and of definite strength.

Dose.—250 milligrammes (4 grains).

EXTRACTUM COLCHICI CORMI. Extract of Colchicum Corm.

Recipe.—Colchicum Corm, in No. 60 powder, *one thousand grammes* 1000 Gm.
Acetic Acid, *three hundred and fifty cubic centimeters*. 350 Cc.
Water, *a sufficient quantity*.

Mix the Acetic Acid with *fifteen hundred cubic centimeters* of Water, and, having moistened the powder with *five hundred cubic centimeters* of the mixture, pack it moderately in a cylindrical glass percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding, first, the remainder of the menstruum, and then Water, until the Colchicum Corm is exhausted. Evaporate the percolate in a porcelain vessel, by means of a water-bath, at a temperature not exceeding 80° C. (176° F.), to a pilular consistence.

When assayed, Extract of Colchicum Corm should contain 1.4 per cent. of colchicine. If the Extract should be found by assay to contain more than this percentage, sufficient powdered sugar of milk should be added to reduce it to the standard of 1.4 per cent.

Remarks.—This is the first recipe for an acetic extract, and differs from that for extract of Indian cannabis in the fact that the menstruum is not the same throughout—first acetic acid and water; then water alone. Note that it is assayed for colchicine, the details of Assay being given in Chapter LV.

Dose.—65 milligrammes (1 grain).

EXTRACTUM COLOCYNTHIDIS. Extract of Colocynth.

Recipe.—Colocynth, freed from the seeds, *one thousand grammes* 1000 Gm.
Diluted Alcohol, *a sufficient quantity*.

Reduce the Colocynth to a coarse powder by grinding or bruising, and macerate it in *thirty-five hundred cubic centimeters* of Diluted Alcohol for four days, with occasional stirring; then express strongly, and strain through flannel. Pack the residue, previously broken up with the hands, firmly in a cylindrical percolator, cover it with the strainer, and gradually pour Diluted Alcohol upon it until the tincture and expressed liquid, mixed together, measure *five thousand cubic centimeters*. Distil off the Alcohol from the mixture by means of a water-bath, evaporate the residue to dryness, and reduce the dry mass to powder.

Extract of Colocynth should be kept in well-stoppered bottles.

Remarks.—In this recipe the drug is first extracted by maceration and expression, then by percolation, the mixed liquids being evaporated to dryness. The extract is used in preparing *compound extract of colocynth* (U. S. P.).

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

EXTRACTUM COLOCYNTHIDIS COMPOSITUM. Compound Extract of Colocynth.

Recipe.—**Extract of Colocynth**, one hundred and sixty grammes..... 160 Gm.
Purified Aloes, five hundred grammes..... 500 Gm.
Cardamom, in No. 60 powder, sixty grammes..... 60 Gm.
Resin of Scammony, in fine powder, one hundred and forty grammes..... 140 Gm.
Soap, dried, and in coarse powder, one hundred and forty grammes..... 140 Gm.
Alcohol, one hundred cubic centimeters..... 100 Cc.

Heat the Purified Aloes, contained in a suitable vessel, on a water-bath, until it is completely melted; then add the Alcohol, Soap, Extract of Colocynth, and Resin of Scammony, and heat the mixture at a temperature not exceeding 120° C. (248° F.), until it is perfectly homogeneous, and a thread taken from the mass becomes brittle when cool. Then withdraw the heat, thoroughly incorporate the Cardamom with the mixture, and cover the vessel until the contents are cold. Finally, reduce the product to a fine powder.

Compound Extract of Colocynth should be kept in well-stoppered bottles.

Remarks.—This, the only official compound extract, is prepared by blending extract of colocynth with aloes, cardamom, resin of scammony, and soap.

This extract contains soap, partly to increase the purgative properties, but chiefly in order that the finished product can be worked into the pill-mass by the addition of water. This explains why, in making compound cathartic pills, water can be used as excipient, the real excipient in this case being the soap contained in the compound extract of colocynth. Used in *compound cathartic pills* (U. S. P.) and in *vegetable cathartic pills* (U. S. P.).

Dose.—500 milligrammes (7½ grains).

EXTRACTUM DIGITALIS. Extract of Digitalis.

Recipe.—**Fluidextract of Digitalis**, one hundred cubic centimeters..... 100 Cc.

Evaporate the Fluidextract of Digitalis in a porcelain dish, by means of a water-bath, at a temperature not exceeding 50° C. (122° F.), with constant stirring, until it is reduced to a pilular consistence.

Remarks.—This recipe—evaporation of the fluidextract—differs from that for extract of cimicifuga in requiring the finished extract to be of pilular consistence, instead of in pulverulent form.

Dose.—10 milligrammes (¼ grain).

EXTRACTUM ERGOTÆ. Extract of Ergot.

Recipe.—**Ergot**, in No. 40 powder, one thousand grammes..... 1000.0 Gm.
Diluted Hydrochloric Acid, fifty grammes..... 50.0 Gm.
Monohydrated Sodium Carbonate, eight and one-half grammes..... 8.5 Gm.
Glycerin, twelve and one-half grammes..... 12.5 Gm.
Alcohol,
Water, each, a sufficient quantity,
To make one hundred and twenty-five grammes.. 125.0 Gm.

Mix one thousand cubic centimeters of Alcohol with four hundred cubic centimeters of Water, and, having moistened the powder with five hundred cubic centimeters of the mixture, pack it firmly in a cylindrical percolator; then add sufficient menstruum

to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding menstruum, using the same proportions of Alcohol and Water as before, until the Ergot is exhausted. Evaporate the percolate in a porcelain dish, by means of a water-bath, at a temperature not exceeding 50° C. (122° F.), to *two hundred and fifty grammes*; add *two hundred and fifty cubic centimeters* of Water, and stir; filter when cold, rinse the dish with a little Water, and add this to the filter. Add the Diluted Hydrochloric Acid to the filtrate; then set it aside for twenty-four hours; filter, wash the contents of the filter with Water until the washings no longer have an acid reaction, and add the washings to the filtrate. To this, add gradually the Monohydrated Sodium Carbonate, and, when the evolution of carbon dioxide has ceased, evaporate the liquid in a tared dish until it weighs *one hundred and fifty grammes*; add the Glycerin, and continue the evaporation, at the above-named temperature, until the weight of the Extract is reduced to *one hundred and twenty-five grammes*.

Remarks.—This extract, like that of belladonna leaves and of Indian cannabis, is prepared by percolation of the drug and evaporation of the percolate. There is, however, a marked difference at this point, by the special treatment of the partially evaporated percolate with hydrochloric acid, which precipitates the inert albumin and holds in solution the active cornutine. The filtered liquid is then neutralized with sodium carbonate, evaporated, and preserved in plastic form by addition of 10 per cent. glycerin.

Dose.—250 milligrammes (4 grains).

EXTRACTUM EUONYMI. Extract of Euonymus.

Recipe.—Made by evaporation of 100 Cc. of the fluidextract to 25 Gm. of dried powdered extract, exactly as in making extract of cimicifuga.

Dose.—125 milligrammes (2 grains).

EXTRACTUM GENTIANÆ. Extract of Gentian.

Recipe.—Gentian, in No. 20 powder, *one thousand grammes*. 1000 Gm.
Water, *a sufficient quantity*.

Moisten the powder with *four hundred cubic centimeters* of Water, and let it macerate for twenty-four hours; then pack it in a conical percolator, and gradually pour Water upon it until the infusion passes but slightly imbued with the properties of the Gentian. Reduce the liquid to three-fourths of its bulk by boiling, and strain; then, by means of a water-bath, evaporate to a pilular consistence.

Remarks.—Made by the evaporation of the aqueous percolate from gentian, this liquid being strained after boiling to three-fourths its original bulk, and then evaporated to pilular consistence. The use of the extract as a pill excipient, so popular in the South, is not to be recommended from the medical point of view (p. 315).

Dose.—250 milligrammes (4 grains).

EXTRACTUM GLYCYRRHIZÆ. Extract of Glycyrrhiza.

The commercial extract of the root of *Glycyrrhiza glabra* Linné, or of *Glycyrrhiza glandulifera* Waldstein and Kitaibel (Fam. Leguminosæ).

In flattened cylindrical rolls, from 15 to 18 Cm. long, and from 15 to 30 Mm. thick; of a glossy black color. It breaks with a sharp, conchoidal, shining fracture, and has a very sweet, peculiar taste. Not less than 60 per cent. of it should be soluble in cold water.

Manufacture.—Prepared on a large scale in Italy by evaporating a strained aqueous infusion in vacuum pans, and rolling the extract while still soft into sticks.

Remarks.—This extract is the commercial product known as “stick licorice.” Samples found in the market are frequently adulterated with starch and other soluble matter; hence the pharmacopœial requirement that at least 60 per cent. be soluble in cold water.

Dose.—1 Gm. (15 grains).

EXTRACTUM GLYCYRRHIZÆ PURUM. Pure Extract of Glycyrrhiza.

Recipe.—Glycyrrhiza, peeled, in No. 20 powder, one thousand grammes..... 1000 Gm.
 Ammonia Water, one hundred and fifty cubic centimeters..... 150 Cc.
 Glycerin,
 Water, each, a sufficient quantity.

Mix the Ammonia Water with *three thousand cubic centimeters* of Water, and, having moistened the powder with *one thousand cubic centimeters* of the mixture, allow it to macerate in a closed vessel for twenty-four hours. Then pack it moderately in a cylindrical glass percolator, and gradually pour upon it, first the remainder of the menstruum, and then Water, until the Glycyrrhiza is exhausted. Lastly, evaporate the liquid in a tared porcelain dish, by means of a water-bath, to a pilular consistence, and, while the mass is still warm, incorporate with it 5 per cent. of its weight of Glycerin.

Remarks.—This extract is prepared by percolating the drug with ammoniacal water, and is the soft extract used in *compound mixture of glycyrrhiza* (U. S. P.). Note that the commercial *purified extract of licorice* is usually in powdered form, hence not identical with the official product.

It will be remembered that ammonia is used in the menstrua of most licorice preparations, owing to the fact that the sweet principle, glycyrrhizin, is most easily soluble in alkaline fluids.

Dose.—1 Gm. (15 grains).

EXTRACTUM HÆMATOXYLI. Extract of Hematoxylon.

Recipe.—Hematoxylon, rasped, one thousand grammes..... 1000 Gm.
 Water, ten thousand cubic centimeters..... 10000 Cc.

Macerate the Hematoxylon with the Water for forty-eight hours. Then boil (avoiding the use of metallic vessels) until one-half of the Water has evaporated; strain the decoction, while hot, and evaporate it to dryness.

Remarks.—This extract is similar to the *extract of logwood* sold in the stores for dyeing purposes. It is not identical, however, for the extract used as dye is prepared from wood which has been allowed to go through a preliminary fermentation process, thereby improving its tinctorial properties.

Dose.—1 Gm. (15 grains).

EXTRACTUM HYOSCYAMI. Extract of Hyoscyamus.

Recipe.—Fluidextract of Hyoscyamus, one hundred cubic centimeters..... 100 Cc.

Evaporate the Fluidextract of Hyoscyamus in a porcelain dish, by means of a water-bath, at a temperature not exceeding 50° C. (122° F.), constantly stirring, until it is reduced to a pilular consistence.

When assayed, Extract of Hyoscyamus should contain 0.3 per cent. of mydriatic alkaloids. If the Extract should be found by the assay to contain more than this percentage, sufficient powdered sugar of milk should be added to reduce it to the standard of 0.3 per cent.

Remarks.—This recipe is identical with that provided for extract of digitalis except that the resulting soft extract must be standardized to contain 0.3 per cent. mydriatic alkaloids, being diluted with sugar of milk if necessary. (Details of assay given in Part V.)

This extract is a constituent of the official *vegetable cathartic pills*.

Dose.—65 milligrammes (1 grain).

EXTRACTUM KRAMERIÆ. Extract of Krameria.

Recipe.—Krameria, in No. 40 powder, one thousand grammes. . . 1000 Gm.

Water, a sufficient quantity.

Moisten the powder with three hundred cubic centimeters of Water, pack it in a conical glass percolator, and gradually pour Water upon it, until the infusion passes but slightly imbued with the astringency of the Krameria. Heat the liquid to the boiling-point, strain, and evaporate the strained liquid, by means of a water-bath, at a temperature not exceeding 70° C. (158° F.), to dryness.

Remarks.—The recipe is quite similar to that for extract of gentian, and yet differs enough to require reproduction of the official recipe.

Dose.—500 milligrammes (7½ grains).

EXTRACTUM LEPTANDRÆ. Extract of Leptandra.

Recipe.—Made by evaporation of 100 Cc. of the fluidextract to 25 Gm. dried powdered extract, exactly as in making extract of cimicifuga. This extract is a constituent of the official *vegetable cathartic pills*.

Dose.—250 milligrammes (4 grains).

EXTRACTUM MALTI. Extract of Malt.

Recipe.—Malt, in coarse powder, not finer than No. 12, one thousand grammes. 1000 Gm.

Water, a sufficient quantity.

Upon the powder, contained in a suitable vessel, pour one thousand cubic centimeters of Water, and macerate for six hours. Then add four thousand cubic centimeters of Water, heated to about 30° C. (86° F.), and digest for an hour at a temperature not exceeding 55° C. (131° F.). Strain the mixture with strong expression. Finally, by means of a water-bath, or vacuum apparatus, at a temperature not exceeding 55° C. (131° F.), evaporate the strained liquid rapidly to the consistence of thick honey.

Extract of Malt should be kept in well-closed vessels, in a cool place.

Remarks.—This popular extract, recognized by the pharmacopœia of 1880, and omitted in the revision of 1890, is now once more given official recognition. Bear in mind this is the soft extract of malt, not the more or less fermented liquid extract of malt prepared by brewers.

Dose.—16 Cc. (4 fluidrachms).

EXTRACTUM NUCIS VOMICÆ. Extract of Nux Vomica.

Recipe.—**Nux Vomica**, in No. 20 powder, *one thousand grammes*,..... 1000 Gm.

Acetic Acid,

Water,

Alcohol,

Sugar of Milk, dried and in fine powder, each, *a sufficient quantity*.

Mix *five hundred cubic centimeters* of Acetic Acid with *thirteen hundred cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it moderately in a cylindrical glass percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding, first, the remainder of the menstruum and then Water, until the percolate passes but faintly imbued with bitterness, and the Nux Vomica is exhausted. Reserve the first *seven hundred and fifty cubic centimeters* of the percolate, and, having heated the remainder to boiling, filter, and evaporate the filtrate to a soft extract; dissolve this in the reserved portion, and add enough Water to make the liquid measure *nine hundred cubic centimeters*. To the liquid thus obtained add *three thousand cubic centimeters* of Alcohol, shake the mixture well, and set it aside for twenty-four hours, with occasional agitation. Filter the liquid through paper, and wash the residue in the filter with a mixture of Alcohol three volumes, and Water one volume, until the washings are only faintly bitter. Evaporate the filtrate and washings in a porcelain dish, on a water-bath, to dryness. Determine, by the method given below, the percentage of strychnine in the dry Extract; ascertain, by calculation, the amount of strychnine in the remainder of the Extract; add to this enough well-dried Sugar of Milk to bring the quantity of strychnine in the final dry Extract of Nux Vomica to 5 per cent. of the total weight; and, when thoroughly powdered and mixed, transfer the Extract to small, well-stoppered vials.

Extract of Nux Vomica, when assayed, should be found to contain 5 per cent. of strychnine.

Remarks.—The intricacies of the above recipe insure an extract free from fat, from albumin, and from much starch. In the process of 1890 the drug was extracted with hydro-alcoholic menstruum, the alcohol driven off, and the watery residue freed from fat by shaking with ether. In the present official process the drug is extracted with acetic acid and water, thus insuring a percolate free from fat. The liquid does, however, contain albumin and starch, and these substances are precipitated from the evaporated percolate by treatment with alcohol. The liquid is freed from the precipitate by filtration, and the filtrate evaporated to dryness and assayed. Enough sugar of milk is then mixed with the dried extract to make a product containing 5 per cent. strychnine. (Details of assay are given in Part V.)

Dose.—15 milligrammes ($\frac{1}{4}$ grain).

EXTRACTUM OPII. Extract of Opium.

Recipe.—**Powdered Opium**, *one hundred grammes*,..... 100 Gm.

Sugar of Milk, recently dried and in fine powder,

Water, each, *a sufficient quantity*.

Rub the Powdered Opium, in a mortar, into a smooth paste with *two hundred and fifty cubic centimeters* of Water; then transfer to a bottle of the capacity of *one thousand cubic centimeters*, wash the mortar with *seven hundred and fifty cubic centimeters* of water in successive portions, and add the washings to the contents of the bottle. Cork the bottle, and shake it vigorously once every two hours during twelve hours. Then filter through a rapidly acting, double filter, and pour water on the magma slowly, until the filtrate passes nearly colorless and only faintly bitter.

Concentrate the filtrate and washings in a tared dish, on a water-bath, until the contents weigh about *two hundred grammes*, and allow the Extract to become cold. Then weigh it accurately, transfer *twelve grammes* to an Erlenmeyer flask having a capacity of about *one hundred cubic centimeters*, and determine in this portion the amount of morphine by the process of assay given (Chapter LV.), using the same quantities of liquids as there directed for *four grammes* of the dry Extract. In another portion of *five grammes* determine the amount of water by drying it in a flat-bottomed dish, at 100° C. (212° F.), until it ceases to lose weight. From the results thus obtained, ascertain by calculation the amount of morphine and of water contained in the remainder of the Extract, add to this enough well dried Sugar of Milk to bring the quantity of morphine in the final dry Extract to 20 per cent., then evaporate the whole to dryness, reduce it to powder, and transfer it to small, well-stoppered vials.

Remarks.—It will be seen that this extract is prepared by the evaporation of an aqueous infusion of the drug and mixing the evaporated liquid with enough milk-sugar to make a dry powdered extract containing 20 per cent. morphine.

It will be noticed that in accomplishing this result one portion of the moist extract is assayed for morphine; while the moisture is estimated in another portion, from these figures—percentage of morphine and percentage of water—can be calculated the amount of sugar of milk that must be added to make a 20 per cent. dried extract, affording a good illustration of the practical application of alligation, as shown in the following hypothetical problem:

Suppose we have an extract containing 25 per cent. moisture and 16 per cent. of morphine; how much sugar of milk must be added to make a drug extract containing 20 per cent. morphine? This is solved as follows:

100 Gm. of the moist extract contains 25 Gm. water and 16 Gm. morphine.

Then 75 Gm. (100–25) of the dried extract will contain 16 Gm. morphine,

and 100 Gm. of the dried extract will contain $\frac{16}{75} \times 16$, or 21.33 Gm. morphine.

Hence the moist extract on drying will contain 21.33 per cent. morphine.

To reduce this 21.33 per cent. extract to the official 20 per cent. extract it will be necessary to add milk-sugar, and the amount can be solved by alligation, viz., to reduce 21.33 per cent. extract to 20 per cent. extract we must add to each 20 Gm. extract enough milk-sugar to make 21.33 Gm., or, in other words, to each 20 Gm. dried extract we must add 1.33 Gm. sugar of milk. Then to each 75 Gm. dried extract, or to each 100 Gm. moist extract (see above), we must add $\frac{16}{75} \times 1.33$ Gm. or 5 Gm. of milk-sugar. Therefore, if we mix each 100 Gm. moist extract with 5 Gm. milk-sugar and then dry, we will have an extract containing 20 per cent. morphine. (Details of assay are given in Part V.)

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

EXTRACTUM PHYSOSTIGMATIS. Extract of Physostigma.

Recipe.—Physostigma, in No. 80 powder, *one thousand grammes*..... 1000 Gm.

Alcohol,

Glycyrrhiza (peeled, Russian), in No. 80 powder,
each, a sufficient quantity.

Moisten the Physostigma with *four hundred cubic centimeters* of Alcohol, and pack it firmly in a cylindrical percolator; then add enough Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding Alcohol, until *three thousand cubic centimeters* of percolate are obtained, or the Physostigma is exhausted. Reserve the first *nine hundred cubic centimeters* of the percolate, and evaporate the remainder, at a temperature not exceeding 50° C. (122° F.), to *one hundred cubic centimeters*; mix this with the reserved portion, and evaporate, at or below the above-mentioned temperature, on a water-bath, to

dryness. Remove *one gramme* of the Extract, and assay this by the process given in Chapter LV.; from the results thus obtained, ascertain by calculation the amount of ether-soluble alkaloids contained in the remainder of the Extract, add to this enough powdered Glycyrrhiza to bring the quantity of the alkaloids in the finished powdered Extract to 2 per cent., reduce to powder, mix thoroughly, and transfer it at once to well-stoppered, amber-colored vials.

Extract of *Physostigma*, when assayed, should be found to contain 2 per cent. of ether-soluble alkaloids.

Remarks.—This extract is made by percolation and evaporation, like extract of belladonna, but the result is a powdered extract, enough powdered glycyrrhiza being added to make the finished product contain 2 per cent. ether-soluble alkaloids. (Details of assay will be given in Part V.)

Dose.—8 milligrammes ($\frac{1}{8}$ grain).

EXTRACTUM QUASSIÆ. Extract of Quassia.

Recipe.—Quassia, in No. 20 powder, *one thousand grammes*. 1000 Gm.

Water,

Sugar of Milk, recently dried and in fine powder,
each, *a sufficient quantity*,

To make *one hundred grammes*.. 100 Gm.

Moisten the Quassia with *four hundred cubic centimeters* of Water, pack it firmly in a conical percolator, and gradually pour Water upon it until the infusion passes but slightly imbued with bitterness. Reduce the liquid to three-fourths of its bulk by boiling, and strain; then evaporate, by means of a water-bath, to dryness and add enough Sugar of Milk to make the Extract weigh *one hundred grammes*. Mix thoroughly, reduce to fine powder, and transfer to well-stoppered bottles.

Remarks.—Like the official extracts of gentian and krameria, this extract is prepared by percolation of drug with water and evaporation of the aqueous percolate. Details of manipulation are different, however, hence the official recipe is reproduced above.

Dose.—65 milligrammes (1 grain).

EXTRACTUM RHAMNI PURSHIANÆ. Extract of Cascara Sagrada.

Recipe.—Cascara Sagrada, in No. 60 powder, *one thousand grammes*..... 1000 Gm.

Alcohol,

Water,

Glycyrrhiza (peeled, Russian), in No. 80 powder,
each, *a sufficient quantity*,

To make *two hundred and fifty grammes*.. 250 Gm.

Mix *one hundred and twenty-five cubic centimeters* of Alcohol with *eight hundred and seventy-five cubic centimeters* of Water, and, having moistened the powder with *four hundred cubic centimeters* of the mixture, pack it firmly in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding menstruum, using the same proportions of Alcohol and Water as before, until the drug is exhausted. Reserve the first *eight hundred and fifty cubic centimeters* of the percolate, and evaporate the remainder on a water-bath, at a temperature not exceeding 70° C. (158° F.), to the consistence of syrup. Mix this with the reserved portion, and continue the evaporation, at or

below the above-mentioned temperature, to dryness. Reduce the Extract to fine powder, and add enough powdered Glycyrrhiza to make the product weigh *two hundred and fifty grammes*. Mix thoroughly.

Remarks.—This recipe closely resembles that for extract of physostigma. It differs, however, in strength of menstruum, amount of reserved portion, temperature of evaporation, and, most important of all, that the drug extract is mixed with enough powdered glycyrrhiza to make a 400 per cent. product. In other words, there is no assay of active constituents, as in the case of extract of physostigma.

Dose.—250 milligrammes (4 grains).

EXTRACTUM RHEI. Extract of Rhubarb.

Recipe.—Made by evaporating the fluidextract to pilular consistence. Details identical with those of the recipe for extract of digitalis.

Dose.—250 milligrammes (4 grains).

EXTRACTUM SCOPOLÆ. Extract of Scopolia.

Recipe.—Made by evaporating the fluidextract to pilular consistence, adding enough sugar of milk (if necessary) to make finished product contain 2 per cent. mydriatic alkaloid. Details identical with those of recipe for extract of hyoscyamus.

Remarks.—Explanation of assay of the extract will be given in Part V.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

EXTRACTUM STRAMONII. Extract of Stramonium.

Recipe.—Identical with that for extract of hyoscyamus, except that the finished product must assay 1 per cent. mydriatic alkaloids. (Details of assay given in Part V.)

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

EXTRACTUM SUMBUL. Extract of Sumbul.

Recipe.—Identical with that for extract of digitalis, except that the temperature of evaporation to pilular consistence is put at 70° C.

Dose.—250 milligrammes (4 grains).

EXTRACTUM TARAXACI. Extract of Taraxacum.

Recipe.—Taraxacum, in No. 30 powder, *one thousand grammes*..... 1000 Gm.

Alcohol,

Water, each, *a sufficient quantity*.

Mix *one hundred and twenty-five cubic centimeters* of Alcohol with *eight hundred and seventy-five cubic centimeters* of Water, and, having moistened the powder with *two hundred and fifty cubic centimeters* of the mixture, pack it in a cylindrical percolator; then add enough menstruum to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for twenty-four hours. Then allow the percolation to proceed, gradually adding menstruum, using the same proportions of Alcohol and Water as before, until the Taraxacum is exhausted. Evaporate the percolate, by means of a water-bath, to a pilular consistence.

Remarks.—While this recipe is like to that of the other extracts prepared by percolating the drug with hydro-alcohol menstruum and evaporation of the percolate, it differs from the others in directing evaporation to pilular consistence without attempt at standardization.

Dose.—1 Gm. (15 grains).

Doses of Official Extracts

8 milligrammes ($\frac{1}{8}$ grain).....	Extract of physostigma.
10 milligrammes ($\frac{1}{8}$ grain).....	Extracts of belladonna leaves, digitalis, Indian cannabis, scopolia, and stramonium.
15 milligrammes ($\frac{1}{4}$ grain).....	Extract of nux vomica.
30 milligrammes ($\frac{1}{2}$ grain).....	Extracts of colocynth and opium.
65 milligrammes (1 grain).....	Extracts of colchicum corm, hyoscyamus, and quassia.
125 milligrammes (2 grains).....	Extracts of aloes and euonymus.
250 milligrammes (4 grains).....	Extracts of cascara sagrada, cimicifuga, ergot, gentian, leptandra, rhubarb, and sumbul.
500 milligrammes (7 $\frac{1}{2}$ grains).....	Extracts of colocynth compound and krameria.
1 gramme (15 grains).....	Extracts of glycyrrhiza, glycyrrhiza pure, hematoxylon, and taraxacum.
16 cubic centimeters (4 fluidrachms).	Extract of malt.

ABSTRACTS

Abstracts were a class of preparations introduced into the pharmacopœia of 1880, intended to supplant extracts, they representing powders containing the active principles of drugs in the condensed form, and of definite strength, prepared by percolating the drug with the appropriate menstruum, reserving a certain portion, say, 90 per cent. of the weight of the drug, evaporating the weak percolate of the drug to a thin extract, blending with the reserve portion and also with sugar of milk; evaporating the mixture at a low temperature until dry. The mass was then weighed, and enough sugar of milk added to make the finished product exactly half the weight of the drug from which it was derived. Such an abstract, therefore, represents twice the strength of the drug.

Abstracts possess over soft extracts the great advantage of being in powdered form and of definite and uniform strength, but, unfortunately, they won little favor from physicians and were dropped in the revision of 1890. Their advocacy was not in vain, however, since in the present pharmacopœia, as already mentioned on p. 278, almost half of the official extracts are in powdered form, 14 of the 28 are standardized, and of the 14, 10 have been given uniform strength, representing about 400 per cent. of the drug.

RESINS

While in the chemical sense resins are solid plant substances or exudations usually acid in character, insoluble in water, and soluble in water and alkali, the definition of the pharmaceutic class—resins—are *those plant products, soluble in alcohol and insoluble in water, obtained either as the residue from the distillation of an oleoresin, or by pouring a concentrated alcoholic extract of a drug into water or acidulated water.* All three official resins are made by the latter process; but, as example of resins obtained by distillation of an oleoresin, may be

cited resin of copaiba (U. S. P. 1890) and official rosin. The latter, however, can best be considered as a crude drug, and is, therefore, discussed in Part IV.

SPECIAL NOTES ON OFFICIAL RESINS

RESINA JALAPÆ. Resin of Jalap.

Recipe.—Jalap, in No. 60 powder, *one thousand grammes*.... 1000 Gm.

Alcohol,

Water, each, *a sufficient quantity*.

Moisten the powder with *three hundred cubic centimeters* of Alcohol, and pack it firmly in a cylindrical percolator; then add enough Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed slowly, gradually adding Alcohol, until *twenty-five hundred cubic centimeters* of percolate are obtained, or until the percolate ceases to produce more than a slight turbidity when dropped into water. Distil off the Alcohol, by means of a water-bath, until the percolate is reduced in weight to *two hundred and fifty grammes*, and add the latter slowly, with constant stirring, to *three thousand cubic centimeters* of water. When the precipitate has subsided, decant the supernatant liquid, and wash the precipitate twice, by decantation, with fresh portions of hot water. After having drained off all the liquid, transfer the Resin to a porcelain dish and heat it to dryness on a water-bath.

Yellow to brown masses or fragments, breaking with a resinous, glossy fracture, translucent at the edges, or a yellowish-gray to yellowish-brown powder, having a slight, peculiar odor, and a somewhat acrid taste. Permanent in the air. Soluble in alcohol in all proportions; insoluble in carbon disulphide, benzene, and fixed or volatile oils. Its alcoholic solution has a faintly acid reaction to blue litmus-paper. Not more than 10 per cent. of Resin of Jalap should be soluble in ether, and not more than 35 per cent. in chloroform.

Slowly but completely soluble in 5 times its weight of ammonia water; when this solution is acidified with hydrochloric acid, only a slight turbidity should appear (absence of *rosin, guaiac*, and *other resins*).

Resin of Jalap should not suffer any material loss of weight when heated at 100° C. (212° F.) (absence of *water*). Anhydrous Resin of Jalap melts at about 150° C. (302° F.).

Water triturated with Resin of Jalap should neither become colored nor dissolve any portion of it (absence of *soluble impurities*).

No greenish-blue color should be produced on adding a few drops of ferric chloride T.S. to some of the powder moistened with alcohol (absence of *guaiac*).

One Gm. of Resin of Jalap when dissolved in 50 Cc. of alcohol containing 1 Cc. of phenolphthalein T.S. should require not more than 0.5 Cc. of half-normal alcoholic potassium hydroxide V.S. to produce a red color (limit of *acid resins*).

If to 1 Gm. of Resin of Jalap, dissolved in 50 Cc. of alcohol in a flask, 25 Cc. of half-normal alcoholic potassium hydroxide V.S. be added, and the mixture be heated on a water-bath for one hour, and if the excess of alkali be titrated with half-normal sulphuric acid V.S., using 5 drops of phenolphthalein T.S. as indicator, at least 20 Cc. of half-normal sulphuric acid V.S. should be required (limit of *saponifiable substances*).

Remarks.—The above recipe—precipitation of resin from the concentrated alcoholic tincture by pouring into water—needs no special comment.

As most pharmacists buy their resins in the open market, and as some of the commercial samples are rankly adulterated, the pharmacopœia gives in the case of all three official pharmaceutical resins tests for identity and for detection of adulterants. Whatever explanation of these tests are needed will be given under the drugs from which the resin is derived in Part IV. A constituent of the official *compound cathartic pills* and *vegetable cathartic pills*.

Dose.—125 milligrammes (2 grains).

RESINA PODOPHYLLI. Resin of Podophyllum.

Recipe.—Podophyllum, in No. 60 powder, *one thousand grammes*..... 1000 Gm.
 Hydrochloric Acid, *ten cubic centimeters*..... 10 Cc.
 Alcohol,
 Water, each, *a sufficient quantity*.

Moisten the powder with *four hundred and eighty cubic centimeters* of Alcohol, and pack it firmly in a cylindrical percolator; then add enough Alcohol to saturate the powder and leave a stratum above it. When the liquid begins to drop from the percolator, close the lower orifice, and, having closely covered the percolator, macerate for forty-eight hours. Then allow the percolation to proceed, gradually adding Alcohol, until *sixteen hundred cubic centimeters* of percolate are obtained, or until the percolate ceases to produce more than a slight turbidity when dropped into water. Distil off the Alcohol, by means of a water-bath, until the percolate is reduced to the consistence of a thin syrup, and pour it slowly, with constant stirring, into *one thousand cubic centimeters* of water, previously cooled to a temperature below 10° C. (50° F.), and mixed with the Hydrochloric Acid. When the precipitate has subsided, decant the supernatant liquid, and wash the precipitate twice, by decantation, with fresh portions of cold Water. Spread it in a thin layer, upon a strainer, and dry the Resin by exposure to the air, in a cool place, protected from the light. Should it coalesce during the drying, or aggregate into lumps having a varnish-like surface, it should be removed, broken in pieces, and rubbed in a mortar.

It should be kept in amber-colored, well-stoppered vials.

An amorphous powder, varying in color from grayish-white to pale greenish-yellow, turning darker when subjected to a heat exceeding 35° C. (95° F.) or when exposed to light. It has a slight, peculiar odor and a faintly bitter taste; very irritating to the mucous membrane, especially to that of the eyes.

Soluble in alcohol in all proportions; not less than 75 per cent. of Resin of Podophyllum should be soluble in ether, not less than 65 per cent. in chloroform, and not more than 25 per cent. in boiling water. A hot, aqueous solution deposits most of its contents on cooling, and if the cooled liquid be filtered, the filtrate has a bitter taste, and turns brown upon the addition of a few drops of ferric chloride T.S. Soluble in potassium or sodium hydroxide T.S., forming a deep yellow liquid, which gradually becomes darker on standing, and from which the Resin is reprecipitated by acids.

Not less than 99 per cent. of Resin of Podophyllum should be soluble in alcohol; the solution should be clear or, at most, slightly opalescent, and should have a faintly acid reaction.

Upon incineration, Resin of Podophyllum should yield not more than 0.7 per cent. of ash.

Remarks.—It will be noted that this recipe differs from that for resin of jalap in amount of alcohol used in moistening drug; in the amount of percolate collected; in volume to which the tincture must be concentrated; in the fact that the water in which the precipitation is performed is acidulated and must be cold, and that the wash-water must be cold.

The question of temperature and composition of the precipitation liquid is of great importance in making this and other resinoids. Some manufacturers precipitate podophyllin by pouring into alum solution, securing a larger yield and a less active product.

This resin is the most important of the so-called "*resinoids*," a class of preparations introduced by eclectic practitioners, through whose efforts a large number of valuable American drugs—such as podophyllum and hydrastis—were brought to the attention of physicians.

In attempting to secure concentrated forms of these eclectic drugs Dr. King hit upon the idea of extracting the drug with strong alcohol and pouring this alcoholic tincture into water. In this way

he obtained resin of podophyllum, and so named it. Later manufacturers, supposing it a definite body, called it "podophyllin."

Like many other excellent ideas, the search for such concentrates was carried to excess, and thus were put upon the market a number of *resinoids*, each purporting to be the active principle of the plant from which derived, and given the name implying such active constituent. Among such resinoids of little or no value may be cited *aletrin* (from *Aletris farinosa*); *geranin* (from *Geranium maculatum*); *macrotrin* (from *Cimicifuga racemosa*, the other botanical name of which is *Macrotrys actæoides*); and *sanguinarin* (from *Sanguinaria canadensis*). How these commercial names lead to confusion can best be shown by the statement that the resinoid, sanguinarin, has nothing in common with sanguinarine, the alkaloid of *sanguinaria*.

Resin of podophyllum is a constituent of the official *vegetable cathartic pills* and *pills of podophyllum, belladonna, and capsicum*.

Dose.—Purgative, 15 milligrammes ($\frac{1}{10}$ grain); laxative, 5 milligrammes ($\frac{1}{20}$ grain).

RESINA SCAMMONII. Resin of Scammony.

Recipe.—Scammony, in No. 60 powder, one thousand grammes. . 1000 Gm.
Alcohol,
Water, each, a sufficient quantity.

Digest the Scammony with successive portions of boiling Alcohol until it is exhausted. Mix the liquids, filter, and reduce the mixture to a syrupy consistence by distilling off the Alcohol. Then add the residue in a thin stream, with active stirring, to twenty-five hundred cubic centimeters of Water, separate the precipitate formed, wash it thoroughly with Water, and dry it at a gentle heat.

Yellowish-brown or brownish-yellow masses or fragments, breaking with a glossy resinous fracture, translucent at the edges; or a yellowish-white or grayish-white powder, having a faint, characteristic odor, and a slight, peculiar taste.

Soluble in alcohol in all proportions, completely soluble in oil of turpentine, and almost completely soluble in ether and chloroform. Ammonia water and solutions of alkalis dissolve it with the aid of a gentle heat, and from these solutions the Resin is not reprecipitated by acids.

When incinerated, it should not yield more than 1 per cent. of ash.

Sulphuric acid, when stirred in a porcelain dish with an equal weight of Resin of Scammony, should not gradually turn red (absence of *rosin*).

Remarks.—Note that in making this resin the drug is extracted by digestion with boiling alcohol, not by percolation.

Dose.—200 milligrammes (3 grains).

RESINA COPAIBÆ (U. S. P. 1890). Resin of Copaiba.

This resin, which was official in former pharmacopœias, but dropped at the last revision, is made by distilling the oleoresin, copaiba, when the volatile oil passes over, leaving the resin in the retort.

Doses of Official Resins

5 milligrammes ($\frac{1}{20}$ grain).....	} Resin of podophyllum.
15 milligrammes ($\frac{1}{4}$ grain).....	
125 milligrammes (2 grains).....	Resin of jalap.
200 milligrammes (3 grains).....	Resin of scammony.

CHAPTER XVIII

POWDERS AND TRITURATIONS

POWDERS

A DEFINITION for powders is somewhat difficult unless we are satisfied with the rather vague statement, *combinations of solid drugs in finely divided form*.

It is even questionable whether powders should be grouped under "internal preparations," for while all official powders are used internally, there are many prescriptions for powders intended for dusting purposes. Powders are usually made by trituration with pestle and mortar, though dusting-powders should be made by mixing the ingredients, already reduced to a fine powder, with a spatula, since rubbing in a mortar is apt to cause caking.

In writing prescriptions for compound powders the physician usually directs the trituration of a sufficient quantity of the ingredients to make several powders. This is particularly useful from the point of the physician in enabling him to prescribe a certain weighable quantity of a potent drug, say, one grain of arsenic, and directing that to be rubbed up with the other constituents, and then to be dispensed in a sufficient number of parts, so that each part will contain a safe dose. Thus, if a grain of arsenic is directed in 30 powders, each powder will contain the usual dose of that chemical ($\frac{1}{30}$ grain). In preparing such powders it is necessary for the pharmacist to exert great caution in having the potent ingredient sufficiently triturated with the diluent to insure uniform and thorough subdivision. The dividing of the mass into the requisite number of powders is accomplished in one of three ways; the papers, which are to hold the several portions of the divided powder, are spread on a clean table; the mixture transferred to the papers in portions by means of the spatula, and when all the substance has thus been transferred, an attempt is made to equalize the quantity of powder on each paper by means of the eye. With practice one becomes fairly expert at striking the requisite quantity, although the process, at its best, is inaccurate, as can be proved by any pharmacist by taking at random three or four powders from the batch that is thus divided and weighing each. A variation of 25 to 30 per cent. is not unusual.

In order to render the subdivision more accurate, several appliances for the dividing of powders have been devised. Of these the most convenient is the Michael powder divider (Fig. 167). This consists of a tapering cup into which the mixed powder is poured and packed fairly uniformly by gentle tapping. Into this is then inserted a special separator consisting of metal segments, which exactly divide the cup into the number of powders desired, thus equally dividing the powder into the desired portions. This being done, the apparatus is covered with a tightly fitting cap, provided with one orifice, through which the contents of compartments of the divider can be poured off one at a time. By turning the cap so that the orifice changes from segment to segment, and inverting after

each case, the contents of each segment can be emptied. At its best, however, such a powder divider is not exceedingly convenient, nor is it so correct as one would wish.

The third, and by all odds the best, method of dividing powders is by estimating the weight that each powder should be, and weighing out that quantity, and transferring it forthwith to one of the unfolded papers. In this way correct results are obtained, and when once practised, the process is found to demand but little more time than that consumed in dividing by eye. In weighing powders it is found, however, by reason of the extreme accuracy of the process, that the last powder is apt to be short in weight, due to the natural loss because of some of the powder adhering to the pestle and mortar. In the writer's practice he always weighed out a sufficient amount of each ingredient called for in the prescription to make one more powder than the number directed, and the slight excess, after weighing out

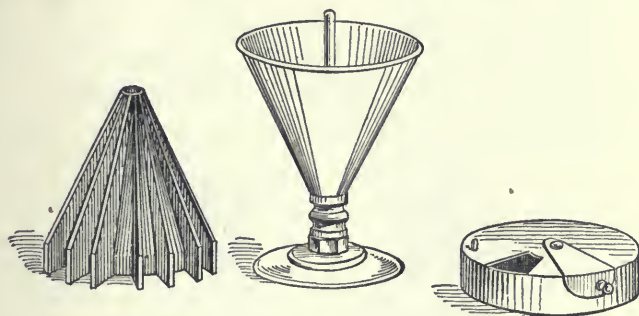


Fig. 167.—Michael's powder divider.

the requisite number of papers, was then thrown away. The powders so divided are then folded in separate pieces of paper; the art of powder-folding being one acquired with a little practice. The process consists in laying the requisite number of papers on the clean table, each provided with a fold at the top, the size of which has been determined in advance. The subsequent processes will best be shown in the appended illustrations (Fig. 168), the second stage being to bring up the lower edge of the paper to the crease already made, as shown in *b*. The flap of the crease is then bent over on to the edge, the paper then taken and creased and folded down to the lower edge of the paper, and then all that remains is that the two edges be folded inward, as shown in *c*. The folding of these ends, so that the length of each folded paper be the same, is sometimes performed on a special powder-folder, shown in Fig. 169, although many careful pharmacists fold the paper as directed upon the box into which the finished powders are to be placed.

There are now on the market powder papers folded by machinery and intended to be used by opening, putting in the powder, and folding again, but the writer fails to see wherein these give better results than a plain paper folded by hand, as directed below.

In discussing the question of the various forms of powder boxes, it is necessary here to say that modern pharmacists use the so-called

New York powder box, shown in Fig. 170, slide powder boxes failing to give as good results. Into this New York powder box the powder should be placed upright, as is shown in Fig. 168, *e*, and in folding the powder each should be exactly the same size, and should project from the box to the same height—usually made about $\frac{1}{16}$ inch. The finer grade of the powder box is arranged with a sufficient difference

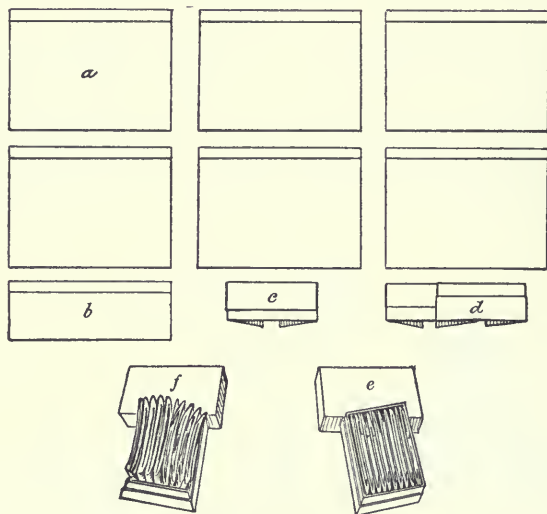


Fig. 168.—Folding powders (see text).

between the height of the shoulder and of the inside of the cover to permit the papers to protrude that distance, the object of having the papers protrude being to facilitate the removing of the papers from the box.

In preparing and successfully folding the paper it is necessary that the paper conform exactly to the dimensions of the box, and



Fig. 169.—Powder folder.

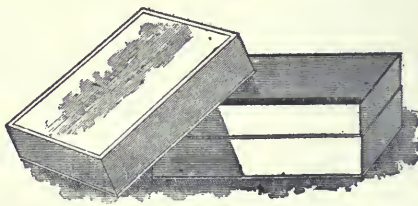


Fig. 170.—New York powder box.

in order to aid the pharmacist in this work, the so-called shoulder boxes are made the same length and height, the difference in the size for the different numbers of papers being merely in the width. In purchasing boxes of that kind the pharmacist should procure paper of such size as will make a perfectly neat package; that is, a paper the length of which is a trifle less than double the interior length of the box, while the width of the paper should be a little over twice the

height of the box. Having obtained paper of the right dimensions, all that remains to be done is to learn the size which the first flap (Fig. 168, *a*) should be made. This is so regulated that the powder, when folded, with the end where the original flap was made coinciding with the finished folded paper, will make a finished powder of the height suggested above, namely, one which will protrude $\frac{1}{16}$ inch from the paper box.

Having folded a paper to conform with the size of the box, it is customary to make the finished powder smooth by rubbing each end down with a spatula. The powders are then gathered up from the counter and piled together with the flaps alternating up and down (Fig. 168, *d*). These precautions are required because it will be seen that the finished powder is thicker where the flap rests than in any other portion of the paper. If the bunch of powders, say 12 to 24, were placed in a box, all in the same position, this slight increase in thickness, multiplied by the number 12 or 24, would make a decided discrepancy in the width of the top and bottom of a bunch of powders; and if such were placed in the box in which they fit snugly, a decided pressure would be exerted at the upper edge, a pressure, in fact, sufficient to cause the springing out of the entire lot of powders when the first paper was removed (Fig. 168, *f*). By alternating the position of the flap up and down the pressure is equalized and no such inconvenience results.

Some pharmacists follow the admirable custom of having their name and address printed on their powder papers in such a position that the printed matter will appear in the center of the folded paper. Such a practice cannot be too strongly recommended, because it affords a highly legitimate method of publicity.

After the powders are neatly folded and placed in the box, the latter is wrapped in an appropriate paper and tied. This leads us to the very important question of the dispensing of packages in elegant form, an art which appears to the writer to be falling into disuse. The first essential in making a successful package is that the paper be of exactly proper proportions, and the writer attributes to the introduction of the roll paper, so largely used in drug-stores at present, and supposed to be unrolled and torn off as required, the lack of care shown in the modern package.

In former days the paper was bought in full sheets and was carefully cut to the size required for the package. Thus, for powder boxes the paper was cut in strips 4 inches wide, and of a length ranging from 6 inches to 8 inches. For pill boxes smaller strips, say $2\frac{1}{2}$ inches wide, were employed. The paper for 1-ounce bottles was 6 by 6 inches, for 2- and 3-ounce bottles were 7 by $8\frac{1}{2}$ inches; for 4- and 6-ounce bottles were $8\frac{1}{2}$ by 11 inches; for 8-ounce bottles was 11 by 11 inches; while for pint bottles it was 12 by 12 inches. Of the papers so cut, the one for 6-ounce bottles usually suffice for the careful wrapping of 1 to 2 ounces of an herb, and from 4 to 8 ounces of a chemical; while the larger sized papers would be used for proportionately larger quantities. The criticism that might be raised in regard to taking pains in making packages is the time which is required. As far as the wrapping of a powder or pill box or of a bottle, no more time is required for wrapping it carefully than is required to do the

same work carelessly, and as for the wrapping of solid substances, it is recommended that the pharmacist follow the custom of old-time druggists of keeping on hand a sufficient stock of packages already wrapped, which can be handed out when demanded. In the author's experience, he kept on hand packages of almost everything for which there was at least one demand a week. Of those substances for which there was a very slight demand, only one package was prepared and kept in the container, and when this was sold, another package was made at the first leisure moment. In this way the pharmacist, especially when by himself in a store, can facilitate dispensing to an enormous extent, because much time is consumed in wrapping packages. Details of making a neat package can be best given accompanied by the following illustrations:

Wrapping a pill box: Take the strip of paper, place the pill box thereon, and bring up the two ends, and fold over into a crease, as is shown in Fig. 171, *a*. The paper should be made of such a size that a second crease about the size of the first one will bring the paper

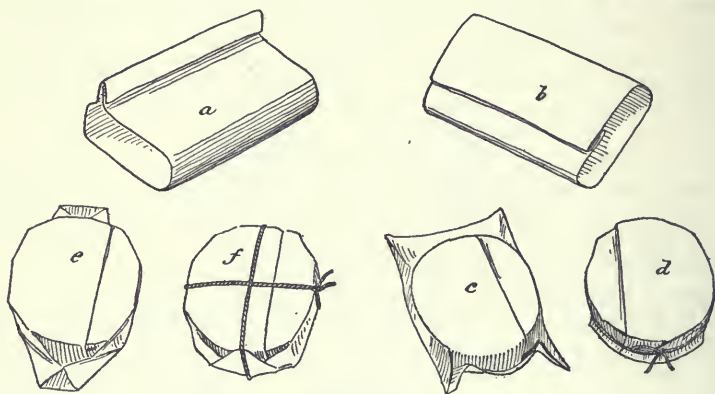


Fig. 171.—Wrapping pill box (see text).

perfectly firmly around the edge of the pill box, as shown in *b*. The paper should then be of sufficient width so that when the edges are folded down in the flap, there will be bulging pieces of paper to be cut off. In the writer's experience, he prefers folding the open edges down to the folded flaps, as shown in *c*, because when so done the tying can be done by simply passing the string two or three times around the edge of the box, as is shown in *d*. Some pharmacists prefer to bring the edges down, as is shown in *e*, when the string must be placed, not merely around the edges, but around the edges of the box, as is shown in *f*. In place of twine, elastic bands are now used, and have much to commend themselves.

In wrapping a powder box the procedure is similar to that of a pill box, as shown in the various stages in Fig. 172, *a* and *b*. The flaps are folded either as shown in *c* or *d*, and for the same reason as explained in the case of the pill boxes.

In wrapping bottles the mode of procedure is, first, to fold the crease, as shown in Fig. 173, *a*, and, as in the case of the powder and pill boxes, the paper should be of such a size that the second fold will

bring the paper snugly around the bottle, as shown in *b*. The open edge at the base of the bottle is then folded in neat flaps, while the paper around the neck should be creased, as shown in *c*.

This done, the bottle is wrapped with string down and back, and finally wound tightly around the neck, as is shown in *d*.

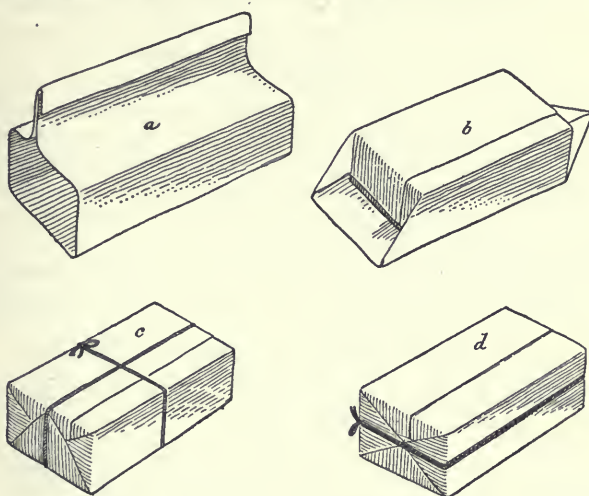


Fig. 172.—Wrapping powder box (see text).

In folding packages the paper is first folded with the crease, as shown in Fig. 174, *a*. This crease is then bent, very great care being required to make it exactly equal the whole length of the package, as shown in *b*. One of the open ends is then temporarily folded,

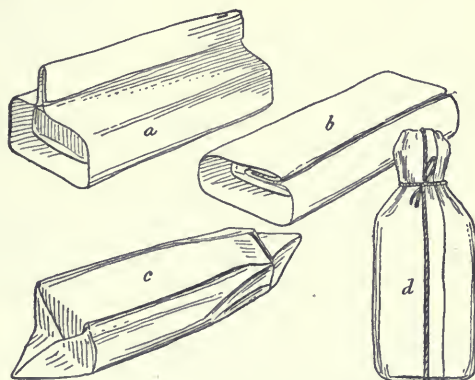


Fig. 173.—Wrapping bottle (see text).

as shown in *c*, while the other end is carefully folded away from the flap, as shown in *d*. This being done, it is then turned downward, the temporary folding of the other end reopened, and refolded exactly as the other end, the finished package then having the appearance as shown in *e*. The package is then wrapped with a string,

both across its length and width, as shown in *e*, although for this purpose a rubber band is frequently substituted.

The folding of solids in packages, as just mentioned, is becoming obsolete, due to the introduction of boxes for holding solid substances at prices justifying their free distribution by the druggist.

Such boxes should be wrapped with paper in a manner similar to the smaller powder boxes, and the use of such boxes is growing very popular because of the convenience of keeping drugs in such containers. It might be here stated that all packages of drugs and chemicals wrapped in the style above given should be neatly labeled.

As to the choice of wrapping-paper, the writer uses plain white paper in preference to the gaudily colored papers so much in vogue in modern pharmacies. He is not alone in this preference, for not long since a woman of refinement and culture spoke of the shocking lack of taste displayed by the average druggist in dispensing his drugs in gaudy pink, blue, and red paper. The manila paper, formerly largely used for the cheaper goods, should not be used by elegant

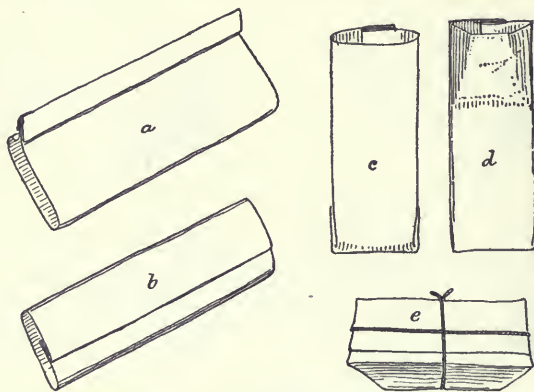


Fig. 174.—Wrapping package (see text).

dispensers; such paper is admirably adapted to the wrapping of packages to be sent by mail or express, but it should not be seen wrapped around any product taken out of the drug-store by the purchaser.

In place of string or rubber bands there has been lately introduced an attractive form of fastener in the shape of ribbon on which is imprinted the name of the firm. Although such ribbon is expensive, the use of it is good business, affording the pharmacist a convenient and unobtrusive method of publicity.

To return to the subject of the dispensing of powders: if the powder contains deliquescent substances, the ordinary powder paper should not be used, as the moisture will soon penetrate to the ingredients and the paper will become wet. Such deliquescent powders should be wrapped in paraffin paper and then covered with tin-foil in order to prevent, as far as possible, atmospheric action.

The *powders of the pharmacopæia* represent mixtures of the medicinal constituents not divided into specific doses, the only exception of this rule being Seidlitz powder.

Even as all the pharmacopœial liquids—such as tinctures and fluidextracts—are directed to be finished as bulk fluids of definite strength, so all official powders except Seidlitz powders are to be finished as bulk masses. In this way the pharmacopœial powders differ from such official solids as pills, lozenges, and suppositories, which are finished in the form of divided doses.

TABLE OF OFFICIAL POWDERS
(ALL GALENIC)

<i>Process.</i>		<i>Preparation.</i>
All by simple trituration. Finished in undivided masses.	Finished	Acetanilid powder, compound. Aromatic powder. Chalk powder, compound. Glycyrrhiza powder, compound. Powder of ipecac and opium. Powder of jalap, compound. Powder of morphine, compound. Powder of rhubarb, compound. Effervescing powder, compound.
Finished in divided doses.		

SPECIAL NOTES ON OFFICIAL POWDERS

PULVIS ACETANILIDI COMPOSITUS. Compound Acetanilide Powder.

<i>Recipe.</i> —Acetanilide, seventy grammes.....	70 Gm.
Caffeine, ten grammes.....	10 Gm.
Sodium Bicarbonate, twenty grammes.....	20 Gm.
To make one hundred grammes..	100 Gm.

Reduce the ingredients separately to a fine powder and mix them thoroughly.

Remarks.—This new official closely simulates the nostrum Antikamnia, which is described on p. 770.

Dose.—500 milligrammes (7½ grains).

PULVIS ANTIMONIALIS (U. S. P. 1890). Antimonial Powder.

This former official is called *James' powder*, and consists of antimony oxide, 33 Gm., triturated with precipitated calcium phosphate, 67 Gm.

PULVIS AROMATICUS. Aromatic Powder.

<i>Recipe.</i> —Saigon Cinnamon, in No. 60 powder, thirty-five grammes.....	35 Gm.
Ginger, in No. 60 powder, thirty-five grammes.....	35 Gm.
Cardamom, deprived of pericarps and crushed, fifteen grammes.....	15 Gm.
Myristica, in No. 20 powder, fifteen grammes.....	15 Gm.
To make one hundred grammes..	100 Gm.

Triturate the Cardamom and Myristica with a portion of the Saigon Cinnamon, until they are reduced to a fine powder; then add the remainder of the Saigon Cinnamon and the Ginger, and rub them together until they are thoroughly mixed.

Remarks.—This consists of cinnamon, ginger, cardamom, and nutmeg. It will be noticed that the cardamom is to be deprived of pericarps (the chaffy wall of ovary constituting the so-called "hull") and crushed, then triturated to a fine powder with coarsely ground nutmeg and a little cinnamon, blended with finely powdered ginger

and cinnamon. In this case it is assumed that the pharmacist will use the freshly grated nutmeg. Both the cardamom and nutmeg are very unstable in the powdered form; hence the whole drugs are to be powdered in the process of preparation.

Aromatic powder is used chiefly to flavor other powdered substances, although it is of some value for relieving flatulence.

Dose.—1 Gm. (15 grains).

PULVIS CRETÆ COMPOSITUS. Compound Chalk Powder.

<i>Recipe.</i> —Prepared Chalk, <i>thirty grammes</i>	30 Gm.
Acacia, in fine powder, <i>twenty grammes</i>	20 Gm.
Sugar, in fine powder, <i>fifty grammes</i>	50 Gm.
To make <i>one hundred grammes</i> ..	100 Gm.

Mix intimately.

Remarks.—This is the chief constituent of *chalk mixture* (p. 264), and is official for that reason alone.

Note that it is made from prepared chalk, not from precipitated chalk, for reasons given on p. 510.

Dose.—2 Gm. (30 grains).

PULVIS EFFERVESCENS COMPOSITUS. Compound Effervescing Powder.

<i>Recipe.</i> —Sodium Bicarbonate, dried and in fine powder, <i>thirty-one grammes</i>	31 Gm.
Potassium and Sodium Tartrate, dried and in fine powder, <i>ninety-three grammes</i>	93 Gm.
Tartaric Acid, dried and in fine powder, <i>twenty-seven grammes</i>	27 Gm.

Mix the Sodium Bicarbonate intimately with the Potassium and Sodium Tartrate, divide the mixture into *twelve equal parts*, and wrap each part in a separate blue paper.

Then divide the Tartaric Acid into *twelve equal parts*, and wrap each part in a separate white paper.

Keep the powders in well-closed containers, in a dry place.

Remarks.—As mentioned previously, this is the one powder in which the constituents are divided into special doses, the sodium bicarbonate and Rochelle salt being divided into 12 powders, wrapped in blue papers, while the tartaric acid is wrapped in white papers. In preparing Seidlitz powders the dividing of the powder is performed by using the so-called Seidlitz powder-cup (Fig. 175); one of the cups is purported to hold the amount of Rochelle salt and sodium bicarbonate mixture, while the smaller cup is supposed to contain the amount of tartaric acid which is directed. These cups are used by taking a large quantity of the powdered substance on a sheet of paper, and filling the cup by pressing into the powdered mass, placing



Fig. 175.—Seidlitz powder cup.

the cup over the powder paper, and emptying on same by a gentle tapping of the cup with a spatula. The use of such cups, however, does not necessarily mean a correct division of the chemicals, as the amount which the cup will hold depends entirely upon the pressure exerted. Some years since an investigator weighed Seidlitz powders

obtained from the various stores throughout the country, and found that the weight of the Rochelle salt—sodium bicarbonate mixture put up in blue paper—ranged all the way from 133 grains to 251 grains, while the white paper contained from 30 grains to 56 grains of tartaric acid. (Proper quantities, 160 grains and 35 grains respectively.)

For this reason it is wisest to weigh out the contents of each powder, a process more feasible than it seems.

The Rochelle salt—sodium bicarbonate mixture—is obtainable in commerce under the name of *Seidlitz mixture*, and that sometimes at a price cheaper than the cost of the separate ingredients. There can be but one explanation of this fact: that such "cheap" Seidlitz mixture contains more than the 25 per cent. of inexpensive sodium bicarbonate directed by the pharmacopœia.

The effervescence occurring on mixing solutions of the contents of the blue and the white papers is due to liberation of carbon dioxide by the chemical action of the tartaric acid on the sodium bicarbonate. Details of this reaction will be found on p. 444.

A difference noted between the average home-made Seidlitz powder and that purchased in the market is the superior keeping quality of acid inclosed in the white papers of the purchased article. Tartaric acid is rather deliquescent, and the home-made "white papers" frequently begin to discolor (the paper wrapper becoming yellow), and not seldom become a moist mass. This trouble can be obviated by thoroughly drying the tartaric acid in an oven or by spreading in the sunshine on a dry day. The tartaric acid so dried will keep as well when wrapped as does that of the commercial Seidlitz powders.

Before leaving the subject of commercial Seidlitz powders reference must be made to an excellent grade found on the market, the subdivision and folding of which are performed on a special machine covered by patents.

This machine—the Doan Seidlitz Powder Machine—is not for sale, the patentee preferring to sell the Seidlitz powders made on the machine, and that at a low price.

Seidlitz powders are much esteemed as purgatives, the effervescence, when administered, masking the taste of Rochelle salts, which is the active ingredient. As is well known, Seidlitz powders are administered by dissolving the contents of the blue and of the white powder in separate glasses one-third filled with water, mixing the two liquids, and drinking while effervescing; *not* by drinking the two liquids one after the other, as has been done more than once, with disastrous results.

Dose.—One set of the powders.

PULVIS GLYCYRRHIZÆ COMPOSITUS. Compound Powder of Glycyrrhiza.

<i>Recipe.</i> —Senna, in No. 80 powder, one hundred and eighty grammes	180 Gm.
Glycyrrhiza, in No. 80 powder, two hundred and thirty-six grammes	236 Gm.
Washed Sulphur, eighty grammes	80 Gm.
Oil of Fennel, four grammes	4 Gm.
Sugar, in fine powder, five hundred grammes	500 Gm.
To make one thousand grammes ..	1000 Gm.

Mix the Oil of Fennel thoroughly with about one-half of the Sugar, then add the remainder of the Sugar and the other ingredients, and mix thoroughly. Finally, pass the powder through a No. 80 sieve, pulverize the residue if any should be left on the sieve, add to the sifted powder, and mix thoroughly. Keep it in well-closed vessels.

Remarks.—Compound powder of glycyrrhiza is what is commonly called “compound licorice powder,” and is an excellent remedy for costiveness. Note its ingredients, and that washed, not sublimed, sulphur is employed.

Dose.—4 Gm. (60 grains).

PULVIS IPECACUANHÆ ET OPII. Powder of Ipecac and Opium.

<i>Recipe.</i> —Ipecac, in No. 60 powder, ten grammes.....	10 Gm.
Powdered Opium, ten grammes.....	10 Gm.
Sugar of Milk, in No. 30 powder, eighty grammes..	80 Gm.
To make one hundred grammes..	100 Gm.

Triturate them together thoroughly and reduce to a very fine powder.

Remarks.—This is the popular *Dover's powder*, and contains 10 per cent. ipecac, 10 per cent. opium, and 80 per cent. sugar of milk.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

PULVIS JALAPÆ COMPOSITUS. Compound Powder of Jalap.

<i>Recipe.</i> —Jalap, in No. 60 powder, thirty-five grammes.....	35 Gm
Potassied Bitartrate, in fine powder, sixty-five grammes	65 Gm.
To make one hundred grammes..	100 Gm.

Rub them together until they are thoroughly mixed.

Remarks.—This powder, sometimes called *pulvis purgans*, consists of 35 per cent. jalap and 65 per cent. cream of tartar.

Dose.—2 Gm. (30 grains).

PULVIS MORPHINÆ COMPOSITUS. Compound Powder of Morphine.

<i>Recipe.</i> —Morphine Sulphate, one and one-half grammes.	1.5 Gm.
Camphor, thirty-two grammes.....	32.0 Gm.
Glycyrrhiza, in No. 80 powder, thirty-three grammes	33.0 Gm.
Precipitated Calcium Carbonate, thirty-three and one-half grammes.....	33.5 Gm.
Alcohol, a sufficient quantity,	
To make one hundred grammes..	100.0 Gm.

Rub the Morphine Sulphate with the Precipitated Calcium Carbonate, added in portions of about five grammes each, until it is thoroughly mixed, then rub the Camphor with a little Alcohol until it is reduced to a powder, and mix intimately with the Glycyrrhiza and the other powders. Finally, pass the powder through a No. 40 sieve, pulverize the residue if any should be left on the sieve, add to the sifted powder, and mix thoroughly. Transfer it to well-stoppered bottles.

Remarks.—This pharmaceutical, called *Tully's powder*, is popular in New England. It contains $1\frac{1}{2}$ per cent. morphine.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

PULVIS RHEI COMPOSITUS. Compound Powder of Rhubarb.

<i>Recipe.</i> —Rhubarb, in No. 60. powder, <i>twenty-five grammes.</i> ..	25 Gm.
Magnesium Oxide, <i>sixty-five grammes.</i>	65 Gm.
Ginger, in No. 60 powder, <i>ten grammes.</i>	10 Gm.
To make <i>one hundred grammes.</i> ..	100 Gm.

Rub the Rhubarb and Ginger together, and, finally, the Magnesium Oxide, gradually added, until they are thoroughly mixed.

Remarks.—Compound powder of rhubarb is yellow when first prepared, but turns pink on standing, due to the action of alkaline magnesia on the resins found in rhubarb.

Dose.—2 Gm. (30 grains).

Doses of Official Powders

500 milligrammes (7½ grains).....	Powders of acetanilid compound, ipecac, and opium; morphine compound.
1 gramme (15 grains).....	Aromatic powder.
2 grammes (30 grains).....	Powders of chalk compound, jalap compound, rhubarb compound.
4 grammes (60 grains).....	Powder of glycyrrhiza compound.
One set of two powders.....	Compound effervescing powder.

TRITURATIONS

Triturations were suggested by similar preparations used in homeopathic practice. The pharmacopœia gives a general formula calling for 10 per cent. of the medicinal substance and 90 per cent. of sugar of milk. There is only one official triturate in which the medicating substance is specifically mentioned, namely, *Triturate of Elaterin*.

TRITURATIONES. Triturations.

Recipe.—Unless otherwise directed, Triturations are to be prepared according to the following formula:

<i>Recipe.</i> —The Substance, <i>ten grammes.</i>	10 Gm.
Sugar of Milk, in moderately fine powder, <i>ninety grammes</i>	90 Gm.
To make <i>one hundred grammes.</i> ..	100 Gm.

Weigh the Substance and the Sugar of Milk separately; then place the Substance, previously reduced, if necessary, to a moderately fine powder, in a mortar; add about an equal measure of Sugar of Milk, mix well by means of a spatula, and triturate the powders thoroughly together. Then add fresh portions of the Sugar of Milk, from time to time, until the whole is added, and continue the trituration after each addition until the Substance is intimately mixed with the Sugar of Milk and reduced to a fine powder.

Remarks.—Note that the sugar of milk must be added little by little.

TRITURATIO ELATERINI. Trituration of Elaterin.

<i>Recipe.</i> —Elaterin, <i>ten grammes.</i>	10 Gm.
Sugar of Milk, in moderately fine powder, <i>ninety grammes.</i>	90 Gm.
To make <i>one hundred grammes.</i> ..	100 Gm.

Mix them thoroughly by trituration.

Dose.—30 milligrammes (½ grain).

OLEO-SACCHARA

This is a class of unofficial preparations which, as suggested by the name, *oleum* and *saccharum*, represents a sugar which has been flavored with some aromatic oil. Such oleo-sacchara are quite popular in Germany, where they are used as diluents in the preparing of many powders. They are made by placing the sugar in a mortar, adding the volatile oil, and triturating until the oil is thoroughly divided by the sugar. The National Formulary gives a general formula for the manufacture of them, directing that they be made of any volatile oil, one drop triturated, with 2 Gm. of sugar. Those mostly prescribed are the oleo-saccharate of anise, of fennel, and of peppermint.

ADMINISTRATION OF POWDERS

A medicated powder is usually administered by placing the proper dose, if small, on the tongue, and then swallowing same in a draft of water. In cases of intensely bitter substances, such administration is scarcely short of inhuman, hence efforts have been made to prevent the bitter substance from coming into contact with the palate by enveloping the drug in a tasteless inert tissue. Such is the origin of the now widely popular cachets and capsules.

CACHETS

The original cachets were wafers made of flour, identical with the consecrated bread used in the sacramental service of the Roman Catholic Church.

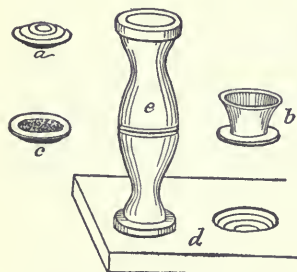


Fig. 176.—Limousin's cachet: *a*, Finished cachet; *b*, cachet funnel (enlarged); *c*, cachet containing powder; *d*, portion of cachet board with (*e*) presser and moistener.

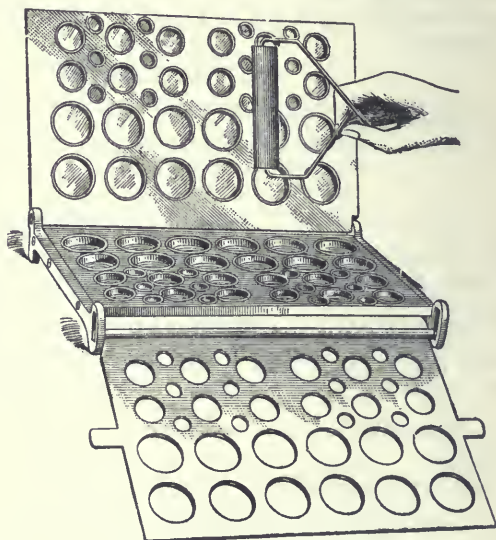


Fig. 177.—Conseal filling apparatus.

These wafer papers were made of pure flour paste, rolled between hot greased rollers, thus driving out the moisture and leaving the paste in a thin, translucent sheet. Such wafer papers are put on the market in either round or square forms, and in dispensing powders

in such wafers the wafer is placed in the bowl of a tablespoon which has been previously moistened with about a half teaspoonful of water. After the wafer has been in the spoon for about a minute, the outer surface will become uniformly moistened, and into the interior of the wafer is then placed the powder, the wafer edges folded over while still in the spoon, thus making a three-cornered or nearly round package. The folded wafer is then quickly immersed in the water, placed in the mouth, and swallowed by aid of a draft of water.

As can be imagined, the administration of powders in wafer papers is exceedingly crude, and it was not long before attempts were made to use the same principle in a more elegant way. This was accomplished by Limousin, who introduced wafers in the form of concave halves, each half being provided with a flange, which can be moistened and pressed to the other half, thus securing adhesion. (See Fig. 176.) The powder is placed on one-half and the other half moistened with water along its flange, placed on the half containing the powder, and in this way a tightly sealed elliptic disk, containing powder on the interior, is obtained. Limousin introduced, along with this cachet, elaborate machinery for the sealing of same, and such a mode for the administration of powders is popular in European countries.

Conseals are a very slight variation of the original Limousin wafer, the shape of them being the same, but the filling apparatus is simpler. The appended sketches fully explain the various portions of the sealing apparatus, so extended comment is unnecessary (Fig. 177).

CAPSULES

Capsules are hemispheric or ovoid shells of gelatin, and are used for the administration of powders, masses, or liquids. According to the quantity of glycerin contained in a capsule mass, the capsules are hard or soft. Hard capsules usually have been the form shown in the appended figures, and consist of a shell and a cover (cap) (*a* and *b* in Fig. 178). Soft capsules are generally shaped like a flask, as

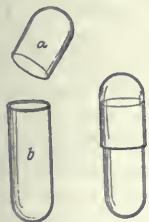


Fig. 178.—Gelatin capsule:
a, The cap; *b*, the body.

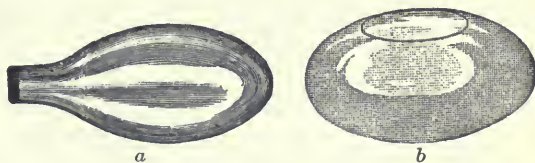


Fig. 179.—Soft capsules: *a*, Unfilled; *b*, filled.

shown in Fig. 179, and these are sealed with a cap by dropping a bit of melted capsule mass on the orifice. The capsules are manufactured by dipping the appropriate mold into the mass, made by dissolving gelatin in a mixture of hot water and glycerin in such proportions that the mass is fluid when heated on the water-bath and solid when cold. Into the melted mass the appropriate mold, made of bone or ivory, is dipped (Fig. 180), with the result of coating each mold with a layer of gelatin. When the molds are taken from the molten mass

and placed in a cool place, they rapidly solidify, and the solid dry capsules are stripped from the molds by girls, who pull them off with their fingers.

Soft capsules are not used to the extent that they should be by the retail pharmacist. They are usually employed for the adminis-

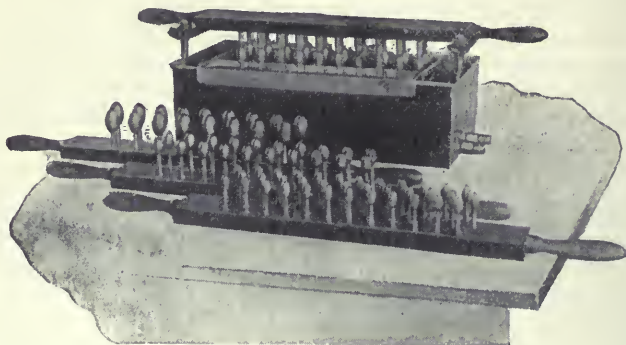


Fig. 180.—Stokes' capsule mold.

tration of liquids, and can be secured of sizes ranging, in capacity, from one fluidrachm to a half fluidounce. The form of soft capsule particularly adapted for dispensing by the pharmacist is that made by the Merz Capsule Co., the firm having also devised an ingenious filler, shown in Fig. 181.

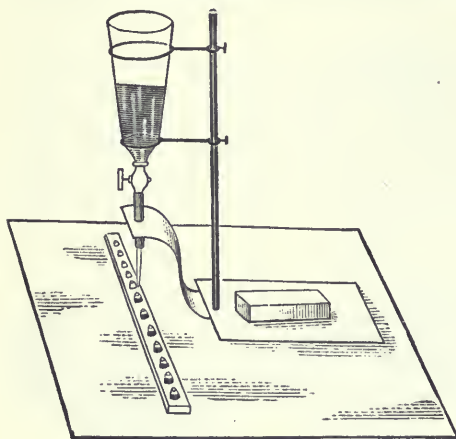


Fig. 181.—Merz capsule filler.

Hard capsules have become a necessity in every drug-store, and, in fact, in almost every household. They range in size from the capacity of 0.12 Cc. (No. 5) to 0.9 Cc. (No. 00); the weight of the medicine they contain, of course, depending on the specific gravity of the substance, and in case of a powder, on the degree of packing. A table of the aver-

age capacity of such capsules for well-known solids is found on the boxes of the best-known brands of capsules.

Such hard capsules can be used for the administration of liquids almost as well as can soft capsules, it being scarcely necessary to state that both hard and soft capsules can be used for holding only those liquids which will not dissolve gelatin. Hence the dispensing of liquids in capsules is limited largely to fixed and volatile oils.

In dispensing oils in hard capsules, the oil is introduced into the capsule by means of a dropper or, better, from a burette; the capsule

is then sealed by moistening sufficiently to cause the cap to adhere to the shell, making a liquid-tight joint.

Two ways of sealing capsules have been suggested, the first by moistening the upper edge of each filled shell with water by means of a delicate camel's-hair pencil which is fastened to the little finger by means of a wire. This process is by no means so satisfactory as the one devised by L. C. Hopp, which consists of placing the caps with the open end down on a flat dish or tin box-cover, into which a minute quantity of diluted alcohol is poured. After letting the caps remain in the diluted alcohol for a minute or two they will have absorbed just enough water to make them adhere and yet not enough to make them soft. After the caps are fitted into the shells, all that is necessary is to rotate that portion of the capsule where the seal has been made between the fingers for a few seconds. Such finished hard capsules, containing oils, should, however, before dispensing, be placed on a filter-paper to see if there is any leak. Those that leak should, of course, be rejected.

In dispensing solid drugs in capsules recourse was formerly had to the working of the solid into a pill mass, dividing the mass into pieces, as in making pills, rolling these pieces into cylinders, and inserting the same into the capsule. Such method of dispensing solids is, however, no longer considered good form, and the pharmacist should always dispense capsules containing the drugs in powder unless otherwise directed by the physician, or, which means the same thing, unless the physician prescribes some semisolid or liquid along with the powdered drug.

Capsules afford a means for dispensing powders in the most readily soluble form, and such capsules containing the powders are

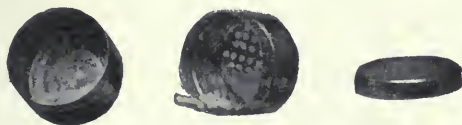


Fig. 182.—Remington capsule cleaner.

much better than any class of pills yet devised, not even excepting the much vaunted friable pills. Such capsules contain nothing but the powders surrounded by a gelatin shell, and the moment they enter the stomach the gelatin dissolves, thus throwing the powders into the stomach in absorbable form. It, therefore, behooves the progressive pharmacist to emphasize to physicians his ability to dispense such capsules and to add the fact that this form of administration of drugs is superior to any form of ready-made pill.

The filling of capsules with powders can be accomplished by placing the mixture of powders on a clean sheet of paper and pressing the shell into the mixture of the powders, when it will gradually fill by gentle pressure. In order to obtain the exact amount of powder directed for each dose, each filled capsule should be weighed, and this is an easy matter by having on one scale the weight plus the empty capsule, and throwing the filled capsule on the other pan. If the exact quantity is not in, a little more pressure will bring the

sufficient weight, and in case of an excess, a slight tapping of the inverted capsule will throw out the surplus. The filled capsule must be carefully cleaned from adhering drug. This can be done by wiping each capsule with a clean dry cloth or, better, by use of the Remington capsule cleaner (Fig. 182).

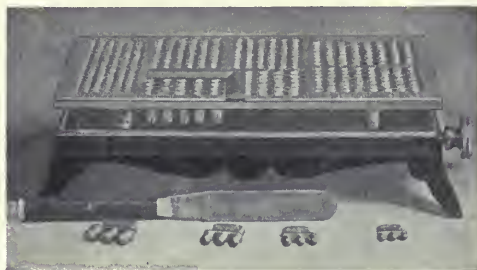


Fig. 183.—Ihrig capsule filler.

There have been a large number of special apparatus for capsule filling placed on the market. Of these, but two forms can here be taken up.

The Ihrig capsule filler is shown in Fig. 183. It consists of a metal base with a movable plate, having perforations to hold the various sized capsules, which can be raised out of the perforations,

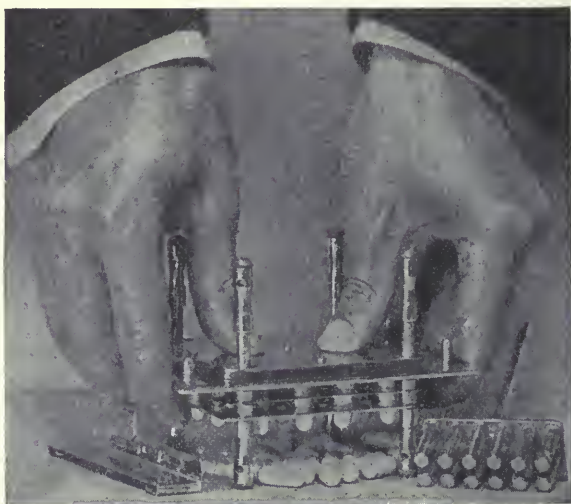


Fig. 184.—Remington automatic capsule filler (see text).

in this case by means of a thumb-screw instead of the inclined plane of an Acme machine. The perforations on the plate are arranged to fill the five sizes of capsules, each set of the different sizes of the perforations being arranged in the portion of the plate by itself. When the apparatus is used, as also in the case of the Acme apparatus, the shell of the capsule should be on a perfect level with the top of the

plate, or a trifle below the surface. When the requisite amount of capsules have been placed in the plate, the powder is poured on that part of the plate and gradually forced into the shell of the capsule by the use of a spatula, the last portion of the work being aided by a punch which fits into three capsules at one time. When all the capsules have been filled, the thumb-screw is turned so that the plate is lowered against the pegs that project from the base, the plugs then pass into the perforations of the plate, thus pushing the shells above the surface of the plate. A cap is then placed on each shell, and the sealed capsule removed from the plate.

The latest filler is the Remington automatic (Fig. 184), which uncaps, fills, recaps, and discharges the capsule with no handling beyond dropping it into the hole.

CHAPTER XIX

MASSES, CONFECTIONS, AND PILLS

MASSES

MASSES are combinations of medicines worked up with enough liquid to make solids of sufficient plasticity to roll into pills.

The manufacture of the official masses is a matter of little difficulty, as in each recipe the quantity of liquid prescribed is sufficient to make a mass of the required plasticity. The manufacture of dry powders into pill masses is a matter requiring skill, and will be fully dealt with when considering pills.

There are but two masses recognized by the pharmacopœia; one of them is a chemical preparation (mass of ferrous carbonate), the other, a galenic (mass of mercury). Mass of copaiba was recognized in the pharmacopœia of 1890, but was dropped at the last revision.

SPECIAL NOTES ON OFFICIAL MASSES

MASSA FERRI CARBONATIS. Mass of Ferrous Carbonate.

This preparation, commonly called *Vallet's mass*, can best be studied among the preparations of iron in Part III.

MASSA HYDRARGYRI. Mass of Mercury.

Recipe and details of manufacture will be found in Part VII.

Remarks.—Mass of mercury is the well-known "blue mass," and its manufacture by hand is a matter demanding skill and labor. The tendency of the mercury to run together and to form globules is well known, and in making the mass we aim to break the mercury into globules so small that they are not even visible under the glass magnifying ten diameters. These minute globules are coated with something that will prevent them from running together, and this process of division, called "extinguishing," is accomplished in the mass by

tritulating the mercury with honey of rose, and then adding althæa, glycyrrhiza, and glycerin.

Dose.—250 milligrammes (4 grains).

MASSA COPAIBÆ (U. S. P. 1890). Mass of Copaiba.

This former official is interesting because 94 parts of the liquid, copaiba, are directed to be solidified by trituration with 6 parts of magnesia; this solidification being due to the formation of the magnesium salt of copaivic acid found in the oleoresin. (See p. 854.)

Every specimen of copaiba, however, does not solidify on treatment with magnesia, nor is it fair to claim that such copaiba is not up to pharmacopœial requirements. The copaibas of commerce are divided into two groups—the ordinary and the solidifiable; and in preparing the mass of copaiba the latter should be used exclusively.

It is claimed, however, that much of the solidifiable copaiba of commerce is made so by addition of ordinary rosin.

There are several proprietary articles on the market under the name of extract of copaiba which closely resemble the official mass. They, however, are usually solidified by trituration with the cheaper alkali, lime.

By reason of the solidification of copaiba with magnesia, that substance is indicated as the excipient in making pills containing copaiba.

CONFECTIONS

Confections are a class of preparations in which medicinal substances are combined with saccharine substances, like jellies or the pulp of fruits. Confections are also called *conserves* and *electuaries*, the word confection being derived from the Latin "*conficio*," meaning to make up. In the days of ancient pharmacy confections were a favorite mode of administration of medicines, since nearly all medicines then administered were nauseous and disgusting, and in order to render them more palatable, attempts were made to cover the unpleasant features by combining them with sweet substances.

In ancient times a large number of confections were used, a famous one of which was the *confection of Damocratis*, which contained from 40 to 50 ingredients.

Instead of the long line of confections of the olden days, but two are recognized by the present pharmacopœia, confection of rose and confection of senna.

CONFECTIO ROSÆ. Confection of Rose.

<i>Recipe</i> .—Red Rose, in No. 60 powder, eighty grammes.....	80 Gm.
Sugar, in fine powder, six hundred and forty grammes.....	640 Gm.
Clarified Honey, one hundred and twenty grammes.....	120 Gm.
Stronger Rose Water, one hundred and sixty cubic centimeters.....	160 Cc.
To make about one thousand grammes..	1000 Gm.

Rub the Red Rose with the Stronger Rose Water previously heated to 65° C. (149° F.), then gradually add the Sugar and Clarified Honey, and beat the whole together until a uniform mass results.

Remarks.—This has practically no medicinal value, but is a pleasant addition to pill masses. No dose is assigned it by the pharmacopœia.

CONFECTIO SENNÆ. Confection of Senna.

<i>Recipe.</i> —Senna, in No. 60 powder, one hundred grammes....	100 Gm.
Cassia Fistula, bruised, one hundred and sixty grammes.....	160 Gm.
Tamarind, one hundred grammes.....	100 Gm.
Prune, sliced, seventy grammes.....	70 Gm.
Fig, bruised, one hundred and twenty grammes.....	120 Gm.
Sugar, in fine powder, five hundred and fifty-five grammes.....	555 Gm.
Oil of Coriander, five grammes..	5 Gm.
Water, a sufficient quantity,	
To make one thousand grammes..	1000 Gm.

Digest the Cassia Fistula, Tamarind, Prune, and Fig with *five hundred cubic centimeters* of Water in a covered vessel, by means of a water-bath, for three hours. Separate the coarser portions and rub the pulpy mass, first through a coarse hair sieve, and then through a muslin cloth. Mix the residue with *one hundred and fifty cubic centimeters* of Water, and, having digested the mixture for a short time, treat it as before, and add the product to the pulpy mass first obtained. Then, by means of a water-bath, dissolve the Sugar in the pulpy liquid, and evaporate the whole in a tared vessel, until it weighs *eight hundred and ninety-five grammes*. Lastly, add the Senna and the Oil of Coriander, and incorporate them thoroughly with the other ingredients while they are yet warm.

Remarks.—Confection of senna is an admirable preparation furnishing an efficient laxative in a palatable form.

A large number of preparations of composition similar to confection of senna have been put on the market as patent medicines, the semisolid mass being broken in pieces and eaten when required.

The best known of this class of patents is *Tamar Indienne*. Of late years even the proprietary articles have fallen into disuse, while the official confection of senna is rarely prescribed by physicians.

Dose.—4 Gm. (60 grains).

PILLS

Pills are spheric, globular, or lenticular masses, each containing a definite dose of medicinal substances, administered by swallowing whole.

The manufacture of pills, while formerly one of the most important branches of pharmacy, is becoming a lost art, due to the introduction of machinery which can perform the work with much greater expedition; but since extemporaneously prepared pills are usually better than the manufactured ones, by reason of the greater solubility, it is important that all the details of their preparations be fully understood.

The manufacture of pills by hand can be roughly divided into three parts: first, making the mass; second, dividing the mass; third, rolling the pills.

Making the Mass.—In performing this operation the medicinal ingredients are, after careful trituration until uniform subdivision is obtained, worked into that plastic semisolid form known as a mass. This mass should possess four requirements if it be ideal: first, it

should be sufficiently *adhesive* that it can be rolled without falling to pieces; second, it should be *firm* enough to retain its shape when rolled, and yet not so soft that the finished pills are flattened during drying; third, it should be *plastic* enough to be easily rolled—that is, not so hard as to make the rolling a matter of considerable labor; fourth, and most important of all, the pill mass should be *soluble*; that is, on being introduced into the stomach it should disintegrate within a reasonable time.

In making a pill mass we usually have recourse to the excipient, and this excipient is different from what we usually and narrowly conceive it to be. Ordinarily, we consider it as a sticky mass with which we “paste” up a solid into pill form. In the broad sense, however, the term applies with equal force to those substances with which we solidify liquids into pill form—like the magnesia used to solidify copal.

Among the excipients used in making pharmaceutical pills are *water*, *sulphuric acid*, *syrup*, *syrup of acacia*, *mucilage of acacia*, *glycerin*, *glucose*, *honey*, *glycerite of tragacanth*, *extract of gentian*, *bread-crumbs*, *soap*, *resin cerate*, *petroleum mass*.

Water is used for making pills only when the pill mass contains some ingredient, which, when moistened, forms an excipient. Thus, if the mixed powder contains gummy substances, say, *acacia* or *soap*, the addition of a few drops of water and working up of the mixture will result in a pliable mass. In such cases, however, the action is the same as if *mucilage of acacia* or *soap* and water were used.

Sulphuric acid, either concentrated or in the form of aromatic sulphuric acid, is sometimes used as an excipient in the manufacture of pills of quinine sulphate. When the acid is added to this chemical it forms a soft and comparatively plastic mass of quinine bisulphate, which can be divided into pills if the mass is worked rapidly. Such method of dispensing quinine sulphate was very largely employed in the South in previous years, but is now falling into disuse.

Syrup is used for certain forms of pills, but is scarcely efficient unless there is present in the mixed powder some gummy substance.

Syrup of acacia would be an excellent excipient were it not for the fact that the finished product, when dry, is usually so hard as seriously to impair its solubility.

Mucilage of acacia has the same advantages and disadvantages of syrup of acacia.

Glycerin is rather extensively used in making the pills of the alkaloids, such as quinine sulphate. It has never been found satisfactory in the hands of the writer by reason of its lack of adhesiveness. One advantage of glycerin as an excipient is that its hygroscopic character keeps the pill soft and thus soluble for quite a while.

Glucose is very largely used as an excipient, being both adhesive and plastic.

Honey is sometimes used, but it has no advantage over glucose, of which it largely consists, and possesses the very decided disadvantage of imparting to the mass a slight brownish tint.

Glycerite of tragacanth, consisting of three drachms of powdered tragacanth, three fluidounces of glycerin, and one-half fluidounce of water, has always been the writer's favorite excipient, it yielding a

mass both plastic and adhesive and firm. Similar results can be gained by the use of the official *glycerite of starch*.

Extract of gentian is frequently prescribed as an excipient and is valuable in tonic pills. *It should, however, never be used as an excipient unless so directed by the physician.* In the first place, gentian has marked medicinal properties, and the pharmacist should never add to a prescription any medicine other than that directed in the recipe. Another point is that it possesses a brownish tint, and in making pills of colorless substances the mass will be discolored, a defect that the careful pharmacist will never permit.

Bread-crumbs are used for holding liquids, such as volatile oils, in the plastic form. Thus, were pill of croton oil directed, the crumb of bread would be indicated. *Magnesia* is used in a similar way with the liquids on which it exerts a solidifying action. Thus, *copaiba* directed in pills should be massed by means of *magnesia* (see above), and this is also true of *Venice turpentine*.

Soap is used in persuading resinous substances to assume the plastic form. Thus, all official pills containing aloes are made plastic and adhesive by the addition of soap. This explains its presence in compound extract of *colocynthis*; and it might here be stated that an attempt to make compound cathartic pills with the simple extract of *colocynthis* instead of the compound extract proves a failure by reason of the absence of soap.

The *resin cerate* and *petroleum mass* are used as excipients for pills containing easily oxidizable substances, such as potassium permanganate, chromic acid, and silver oxide. For this purpose a petroleum mass consisting of kaolin, 3 parts; paraffin, 1 part; and vaselin, 3 parts, can be used to advantage.

The following drugs, frequently prescribed in pill form, require special manipulation:

Creosote, or *guaiacol*, or other oily liquids may be massed by diluting with an absorbent powder like *glycyrrhiza* or *althæa*, and massing with ordinary excipient. From such pills, however, the oils gradually ooze out, and the best way is to inclose the mass in capsules. T. M. Pratt found that by rubbing the oil with *acacia*, then with the diluting powders, and then with excipient, the oil is partially emulsified and the "sweating" is prevented.

Heavy salts, like *bismuth subnitrate*, *calomel*, or *sodium bicarbonate*, can be massed with *glycerite of tragacanth*. If desirable, a small quantity of powdered elm bark may be added.

Bismuth subnitrate and *sodium bicarbonate* when massed together swell and disintegrate, due to liberation of carbon dioxide. The powders should be moistened first with water and let stand, until the reaction is over (one to three hours); then massed with *glycerite of tragacanth*.

The making of a pill mass is performed by thoroughly blending the powders by triturating in a mortar, adding the necessary quantity of excipient, and working the mixture with a pestle until a plastic mass results. (For details of the process see p.1106.) The making of a successful pill mass is merely a matter of practice, and depends largely on the adding of the exact quantity of excipient when first starting the kneading. This really explains the psychology of the

"favorite excipient," it being the writer's experience that the druggist holds as firmly to his pet excipient as he does to his political creed. The reason for this is that the individual has mastered the peculiarities of this special excipient, and such excipient is in his case the best that he can use, inasmuch as he has learned by intuition the exact amount required for each pill mass. Hence, unless the favorite excipient possesses marked defects, such as rendering the pills insoluble or the like, or bringing into the mass a medicinal effect not expected by physicians, no attempt should be made to win a pharmacist from his favorite excipient.

Dividing the Mass.—When the mass has become sufficiently worked, that is, when the pressing of the pestle is sufficient to enable the mass to be rolled from the side of the mortar, it is then rolled into cylinders and divided either on the pill tile or by means of the pill cutter. The details of the use of a pill tile, both in working up the mass and also in dividing the same, is fully described on p. 1106, and it is necessary here only to insert a picture of the apparatus (Fig. 185), which is made of porcelain, with the painted graduations burnt in during the process of glazing. In rolling out the pill mass into cylinders a spatula is used or, what is preferable, a flat board shaped like

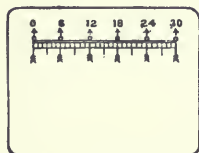


Fig. 185.—Pill tile.

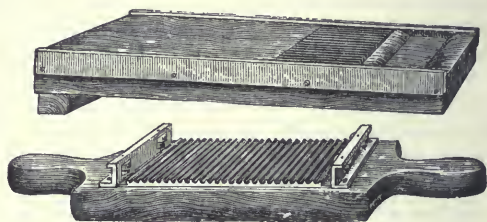


Fig. 186.—Pill-cutting machine.

a paddle. As mentioned on p. 1106, the pill tile should be carefully dusted with the appropriate dusting-powder.

As to pill-cutting machines, these usually consist of brass-bound walnut boards, holding at one end a brass mold, which is divided into a definite number of perfectly uniform hemispheric concave grooves. Working on this is a cutter, which consists of a narrow piece of walnut, provided at each end with stable handles, having attached to one side a brass plate with hemispheric grooves, exactly fitting those on the other part of the apparatus. A set of brass rollers, worked on the brass bands of the sides of the plate, completes the cutter (Fig. 186). In using such a machine the cylinder is placed on the wooden part of the board and rolled out, preferably with a wooden paddle, rather than with the back of cutter, to a length including the number of grooves representing the number of pills desired. The cylindric pill mass is then placed on the grooves of the machine, and the grooves of the cutters are then brought down upon it, thus dividing the cylinder into the number of segments desired. If the diameter of the cylinder exactly coincides with the diameter of the grooves, these segments can be rolled into pills by a gentle rolling of the two plates on each other, and in this way the machine does not merely cut the cylinder, but also rolls them into pills. Special pill machines are on

the market in which the grooved plates are removable from the boards and also from the cutter; and with each machine goes a series of these plates coinciding with the average size of pills. Thus, there is one set of plates for 1-grain pills, 2-grain pills, and 3-grain pills, and one for 5-grain pills. Such machines, however, are rather expensive, and in the average pharmacy the machine is used for cutting rather than for rolling the pills. In purchasing a machine the pharmacist is advised to secure one with plates having at least 36 grooves; the smaller machines, cutting only 12 pills, cost but little less than the wider ones and are not so useful.

Rolling the Pills.—The cut segments, unless rolled in the machines, are taken up between the finger and the thumb and rolled by means of a rotating movement until perfectly spheric. The finishing touches are performed, especially when the mass is hard and difficult to roll between the finger and the thumb, by placing the partially rolled pills on the board of the pill machine, dusting fairly liberally with the dusting-powder, covering with a wooden pill roller (Fig. 187), which is then held firmly by the hand and moved over the board, either in the form of circles or in figure eights, the friction between the roller and the board being sufficient to smooth the pills into spheric forms, providing the mass is sufficiently firm. If the pills are soft the pressure exerted must be very gentle, and it is usually



Fig. 187.—Pill roller.

advisable, in securing successfully rolled pills, that the depth of the roller be exactly the same as the height of the finished pill. To accomplish this, however, it would be necessary to have a set of rollers of different depths, and this is rarely the case in the average pharmacy.

As mentioned on p. 1106, both in rolling and dispensing the pills they are dusted with powder, designed to prevent the pills from sticking together. It is considered good pharmacy to dust the colorless pills with a white powder, such as a finely powdered starch; while those which are of a dark color are usually dusted with lycopodium powder; glycyrrhiza is used as a dusting-powder, especially in Germany, but is not considered so satisfactory as lycopodium. In dispensing ready-made coated pills no dusting-powder should be used.

The finished pills are usually dispensed in round boxes, commercially called pill boxes. Of late years square-shouldered boxes have come into use among the better pharmacies, and are very satisfactory, although the increased cost over the round pill boxes renders their use less popular than the latter.

The wrapping of a pill box has already been described on p. 298. Pills are now made on an enormous scale by machinery, the finest type of which is that made by Arthur Colton, of Detroit. The outfit consists of a kneading machine, which prepares the mass; a rolling machine, in which lumps of the mass are rolled into cylinders; a cutting and finishing machine, which cuts the cylinder into the individual pills and rolls them until spheric or ovoid, as desired. The pills are then dried and coated.

Friable pills are a class introduced by Dr. Upjohn in 1885, and were much vaunted as pills made without excipient and, therefore, capable of disintegrating as soon as the coating is dissolved. The

process of manufacturing these pills is exceedingly clever, consisting of placing the mixed powder containing the medicinal substances in a cylindric pill-coating machine (see p. 319), spraying with water, and introducing therein a lot of sugar pellets, about $\frac{1}{4}$ grain in size. As these pellets are rolled in the moistened powder they gradually collect the powder in a way quite similar to making a large snowball by rolling a small one in damp snow. The rotation of the pills is continued until most of the pills are of the desired size. They are then removed from the machine, and are sifted through special colanders with orifices of exactly the size of the pill desired. Thus, in making quinine pills by this method colanders are provided with holes which have been carefully determined as representing 1-grain, 2-grain, 3-grain, and 5-grain pills. Placing the pills of the various sizes into, say, the 3-grain colander, those remaining on the colander weigh over three grains, while those falling through are from 1 to 3 grains. By placing those which have fallen through on a 2-grain colander, those smaller than 3 grains pass through, and at last the desired size is obtained. The pills which are smaller than any of the required sizes are returned to the machine, and rotation continued until brought up to the weight desired. The finished pills are rapidly coated with either sugar or gelatin, and are thus dispensed.

C. S. N. Hallberg called attention to the fact that the friable pills presented the same objection as is made to most of the commercial pills—that is, in order to obtain a pill the coating of which will not discolor, the rolled pill is first coated with a layer of plaster of Paris and then with the proper coating.

Coating of Pills.—As mentioned above, the fresh pills prepared by the pharmacist are coated merely by dusting with some powder, say, starch or lycopodium. A century ago attempts were made to present the pill in a more attractive appearance by use of special and permanent coating; the first material used for this purpose was



Fig. 188.—Pill siverer.

French chalk, which is a variety of powdered soap-stone (silicate of magnesium). The coating of pills by this process—called *pearl coating*—consists of placing the finished pill in a small dish containing alcoholic or ethereal solution of balsam of tolu, thus coating it with enough of the balsam to be thinly varnished. While this varnish is still wet they are thrown into French chalk, the mixture of pills and chalk placed in a small sieve, and an excess of powder removed by sifting. The pills are then placed in a hollow sphere (Fig. 188), the two hemispheres of which were united by means of a screw-joint. The pills were rotated in this ball by vigorous agitation, and in this way the excess of chalk was thrown off and that remaining was worked into a smooth, pearly coating. The process of silver or gold coating pills was performed in a manner quite similar, the pills were first varnished, as in the pearl coating, then placed in the hollow globe, containing sheets of gold- or silver-foil; by rotating the pills the foil adheres to the same, and by vigorous agitation of the globe the coating became uniform and polished.

Sugar coating was first introduced by confectioners for the purpose of coating almonds. Its use in this way was so successful that there soon arose a demand for pills coated in the same manner, and with this demand began the industry of the manufacture of sugar-coated pills.

The process of sugar coating pills is performed in spheric pans, continuously rotated by means of a central axis by means of steam power. This pan is usually of copper, and is jacketed so that it can be kept uniformly warm with steam. Within the pan a quantity of syrup, almost ready to crystallize, is placed, and when the walls of the pan have become smeared with the syrup, the rounded, dried, uncoated pills are placed therein. As the apparatus rotates, the heat gradually drives off the water from the syrup, with the resulting formation of the layers of sugar on the pills, the process being continued until each pill is uniformly coated with a hard, white, sugary layer. The coated pills are then taken from the apparatus and are rotated in a similar apparatus containing paraffin and lined with rough canvas, when the friction, plus the wax, produces a fine polish.

As mentioned above, the great objection to the sugar-coated pills is the fact that, in order that the coating of pills made of dark constituents does not discolor, it is necessary to coat the pill with a layer of plaster-of-Paris, which certainly does not increase the rapid solubility of the pill. Of late, manufacturers have been placing on the market chocolate and pink-coated pills. These are prepared in a manner similar to the white-coated pills, with the exception that in the first case a small amount of chocolate is added to the syrup, while in the case of the pink, carmine coloring is added to the syrup.

The sugar coating of pills is a process which cannot be done by the retail pharmacist, the coating pan requiring steam power and steam heat. Several schemes of hand-made sugar-coated pills have been suggested, but when tried by the writer, have never yielded satisfactory results. As the public is now demanding sugar-coated pills, the art of pill-making is rapidly falling into disuse.

Gelatin coating of pills is done just as well by the retail pharmacist as by the manufacturer, and several varieties of gelatin-coating apparatus are now on the market at moderate prices. Essentially, the process consists of impaling each pill on a long needle and dipping the same into a solution of gelatin of the proper consistence. Such a solution consists of gelatin, 200; macerate with cold water, 200; then add hot water, 640; mucilage, 300 (acacia, 102; water, 300); syrup, 400; glycerin, 60. Dissolve by gentle heat and then strain. The apparatus devised for the purpose consists merely in carrying out this idea with greater expedition, the needles being arranged in groups, which make it convenient to handle considerable quantities of pills at one time, and also to dry them easily.

Two types of gelatin-coating machines are shown in Figs. 189, 190—the Patch machine and the Maynard machine. In the Patch machine the pills are impaled on the teeth of a comb (*a*); the ingenious part of the process is the drying apparatus, which consists of a wheel holding seven such combs, rotating by means of a string (Fig. 189). In the Maynard machine the needles are grouped on a circular disk, corresponding to the depressions in a circular plate

which holds the pills while being impaled. The impaled pills are then dipped into the gelatin solution and are dried by rapidly whirling the disk holding them. When the coating is dry, the pills are loosened from the pins by pressing down a special plate in the twirler (Fig. 190).

The manufacture of pills by this process yields an admirable gelatin coating, which, however, has been criticized by reason of the

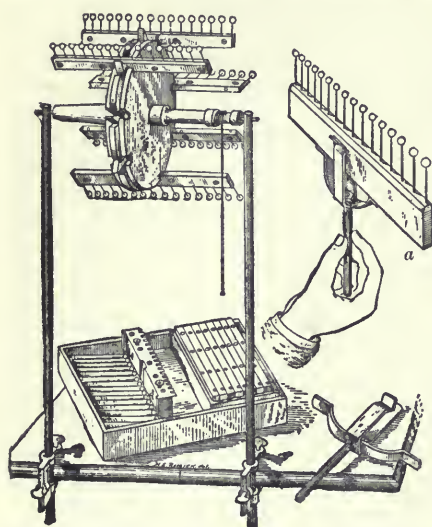


Fig. 189.—Patch's improved gelatin pill-coater.

fact that they contain minute orifices—the pinholes. The presence of this pinhole can in no way interfere with the finished pill, and, if anything, will aid in solubility. But this so-called defect has been seized as an opportunity of manufacturing a pill in which the pinhole would be avoided. This is accomplished by means of the Russell gelatin-coating machine. In this ingenious apparatus the pills, instead of being impaled on pins, are held on the end of minute tubes by means of suction. As shown in Fig. 191, the apparatus consists of a hollow steel plate, the flat sides of which are provided with a series of minute tubes. Suction is applied by means of flexible

tubing connecting the plate with an air-pump, when the plate is held over a disk containing hemispheric depressions exactly corresponding in number and position to the tubes in the plate. In each depression is mechanically rolled a pill, and the moment the plate is placed over the pills, they are sucked to the ends of the tubes and are thus firmly

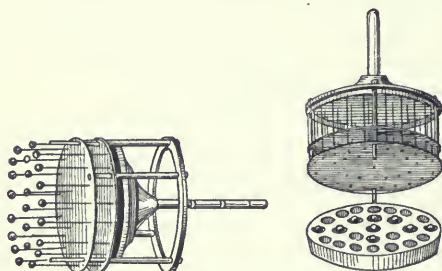


Fig. 190.—Maynard's gelatin coater.

held. The pill is then dipped into the gelatin solution, coated half way, turned upward, and allowed to dry, the stop-cock at the end of the plate being closed, thus continuing the vacuum in the plate even after the rubber tube connected with the air-pump has been removed.

The tubing is then connected with another plate of the same kind, and another set of pills are coated; and in this way the coating is continued until a number of such plates have been filled with the pills and half coated. The plates containing half-coated pills have, in the meanwhile, been placed on a special apparatus consisting of an endless chain carrier, and have been gradually moving toward the other end of the frame, where they are taken charge of by the second operator, who transfers the pills to a second plate by suction in such a way that they are grasped by their coated ends, which are now dry. The pills are then dipped again into the gelatin solution, and the second half of the coating thus completed. The pills are then placed once more on the endless chain carrier, and by the time they reach the other end of the frame they are thoroughly coated and perfectly dry (Fig. 191).

Enteric pills are a special form of pill which are intended to pass through the acid juices of the stomach without dissolving, to dissolve



Fig. 191.—Russell's gelatin coater.

in the alkaline juices of the small intestine. To accomplish this result it is necessary to coat such pills with a material which dissolves in alkalis and not in acids, and for this purpose keratin or salol is employed. Keratin is a commercial product obtained from goose-quills, and the constituent of all horny matter. In using it as a pill coating keratin is dissolved in ammoniacal solution, in alcohol, or in glacial acetic acid. The pill mass must be made with butter of cacao, or oil of sweet almond, and must be free from moisture, and then rotated in the keratin solution until thoroughly moistened, taken out and dried, and the coating repeated three or four times. By reason of the complexity of thus coating pills with keratin it has been suggested to coat with salol by dipping the pills into the melted chemical. The pinhole in both cases of coating must be closed by the addition of a small quantity of coating material. Neither form of enteric pill is very largely used, and it is said that the salol coating does not act so satisfactorily as does keratin.

TABLE OF OFFICIAL PILLS

<i>Process.</i>	<i>Excipient.</i>	<i>Galenic Preparations.</i>	<i>Chemical Preparations.</i>
Massing and rolling. Uncoated.	Water.	Compound pills of rhu- barb.	
	Diluted alcohol.	Pills of aloes and mastic. Compound cathartic pills. Vegetable cathartic pills.	
	Syrup.	Pills of aloes and myrrh. Compound laxative pills.	
	Soap and water.	Pills of aloes. Pills of asafetida. Pills of opium.	
	Confection of rose.	Pills of aloes and iron.	
	Acacia, glycerin, and syrup.	Pills of podophyllum, bel- ladonna, and capsicum.	
Coated pills.	Tragacanth, glycerin, and water.		Pills of ferrous carbonate.
	Acacia and water.		Pills of ferrous iodide.
	Acacia, glycerin, and water.	Pills of phosphorus.	

SPECIAL NOTES ON OFFICIAL PILLS

PILULÆ ALOES. Pills of Aloes.

<i>Recipe.</i> —Purified Aloes, in fine powder, thirteen grammes..	13 Gm.
Soap, in fine powder, thirteen grammes.....	13 Gm.
Water, a sufficient quantity,	
To make one hundred pills..	100

Mix the powders intimately, then incorporate sufficient Water to form a mass, and divide it into *one hundred pills*.

Dose.—Two pills.

PILULÆ ALOES ET FERRI. Pills of Aloes and Iron.

<i>Recipe.</i> —Purified Aloes, in fine powder, seven grammes....	7 Gm.
Exsiccated Ferrous Sulphate, seven grammes.....	7 Gm.
Aromatic Powder, seven grammes.	7 Gm.
Confection of Rose, a sufficient quantity,	
To make one hundred pills..	100

Mix the powders intimately, then incorporate sufficient Confection of Rose to form a mass, and divide it into *one hundred pills*.

Dose.—Two pills.

PILULÆ ALOES ET MASTICHE. Pills of Aloes and Mastic.

<i>Recipe.</i> —Purified Aloes, in fine powder, thirteen grammes...	13 Gm.
Mastic, in fine powder, four grammes.	4 Gm.
Red Rose, in fine powder, three grammes.....	3 Gm.
Diluted Alcohol, a sufficient quantity,	
To make one hundred pills..	100

Mix the powders intimately, then incorporate sufficient Diluted Alcohol to form a mass, and divide it into *one hundred pills*.

Remarks.—These pills are commonly called *Lady Webster's after-dinner pills*. They are massed with diluted alcohol.

Dose.—Two pills.

PILULÆ ALOES ET MYRRHÆ. Pills of Aloes and Myrrh.

<i>Recipe.</i> —Purified Aloes, in fine powder, thirteen grammes...	13 Gm.
Myrrh, in fine powder, six grammes.....	6 Gm.
Aromatic Powder, four grammes.....	4 Gm.
Syrup, a sufficient quantity	

To make one hundred pills.. 100

Mix the powders intimately, then incorporate sufficient Syrup to form a mass, and divide it into one hundred pills.

Dose.—Two pills.

PILULÆ ASAFÆTIDÆ. Pills of Asafetida.

<i>Recipe.</i> —Asafetida, twenty grammes.....	20 Gm.
Soap, in fine powder, six grammes.....	6 Gm.
Water, a sufficient quantity,	

To make one hundred pills.. 100

Beat the solids together with Water, so as to form a mass, and divide it into one hundred pills.

Dose.—Two pills.

PILULÆ CATHARTICÆ COMPOSITÆ. Compound Cathartic Pills.

Recipe and full details of manufacture of these pills will be found in Part VII.

Remarks.—These pills contain compound extract of colocynth, mild mercurous chloride (calomel), resin of jalap, and gamboge, made into a mass with diluted alcohol.

Dose.—Two pills.

PILULÆ CATHARTICÆ VEGETABILES. Vegetable Cathartic Pills.

<i>Recipe.</i> —Compound Extract of Colocynth, sixty grammes	60 Gm.
Extract of Hyoscyamus, thirty grammes.....	30 Gm.
Resin of Jalap, in fine powder, twenty grammes..	20 Gm.
Extract of Leptandra, fifteen grammes.....	15 Gm.
Resin of Podophyllum, fifteen grammes.....	15 Gm.
Oil of Peppermint, eight cubic centimeters.....	8 Cc.
Diluted Alcohol, a sufficient quantity,	

To make one thousand pills.. 1000

Mix the Compound Extract of Colocynth intimately with the Resin of Podophyllum, Resin of Jalap, and Extract of Leptandra, and then add the Oil of Peppermint. Rub the Extract of Hyoscyamus with enough Diluted Alcohol to render it plastic, then incorporate it with the mixture first prepared, using a sufficient quantity of Diluted Alcohol to form a mass, and divide it into one thousand pills.

Remarks.—The pills are similar in action to the compound cathartic pills, but are free from calomel.

Dose.—Two pills.

PILULÆ FERRI CARBONATIS. Pills of Ferrous Carbonate.

These pills, commonly called *Blaud's pills*, depending for their activity upon a chemical action, can best be discussed among the iron preparations in Part III.

PILULÆ FERRI IODIDI. Pills of Ferrous Iodide.

These pills are one of the two official pills directed to be coated with balsam of tolu to prevent oxidation.

Since their manufacture depends on a chemical action, they will be discussed among the iron preparations in Part III.

PILULÆ LAXATIVÆ COMPOSITÆ. Compound Laxative Pills.

<i>Recipe</i> .—Aloin, one and three-tenths grammes.....	1.30 Gm.
Strychnine, five-hundredths of a gramme.....	0.05 Gm.
Extract of Belladonna Leaves, eight-tenths of a gramme.....	0.80 Gm.
Ipecac, in fine powder, four-tenths of a gramme...	0.40 Gm.
Glycyrrhiza, in fine powder, four and six-tenths grammes.....	4.60 Gm.
Syrup, a sufficient quantity,	
To make one hundred pills..	100

Triturate the Aloin, Strychnine, Ipecac, and Glycyrrhiza together thoroughly, so as to produce a uniform powder. Incorporate with this the Extract of Belladonna Leaves and sufficient Syrup to form a mass, and divide it into *one hundred pills*.

Remarks.—This new official has been a most popular laxative during the past decade, and is familiarly known as A. B. and S. pills; it is similar to the commercial *lapactic pills*.

Dose.—Two pills.

PILULÆ OPII. Pills of Opium.

<i>Recipe</i> .—Powdered Opium, six and one-half grammes.....	6.5 Gm.
Soap, in fine powder, two grammes.....	2.0 Gm.
Water, a sufficient quantity,	
To make one hundred pills..	100

Mix the powders intimately, then incorporate sufficient Water to form a mass, and divide it into *one hundred pills*.

Remarks.—Each pill contains about one grain of opium, and is made with soap and water as the excipient.

Dose.—One pill.

PILULÆ PHOSPHORI. Pills of Phosphorus.

Recipe and details of manipulation will be found in Part VII.

Remarks.—Each pill contains about $\frac{1}{100}$ grain of phosphorus, made by mixing a chloroformic solution of phosphorus with althæa and acacia, adding the excipient, glycerin and water, and rapidly massing, cutting, and rolling into pills. The massing must be done rapidly, as phosphorus long exposed to air takes fire spontaneously. The ignition is due to the oxidation of phosphorus, and to prevent this oxidation in the finished pills the pharmacopœia directs that they be coated with balsam of Tolu.

Dose.—One pill.

PILULÆ PODOPHYLLI, BELLADONNÆ ET CAPSICI. Pills of Podophyllum, Belladonna, and Capsicum.

<i>Recipe.</i> —Resin of Podophyllum, <i>one and six-tenths grammes.</i>	1.6 Gm.
Extract of Belladonna Leaves, <i>eight-tenths of a gramme</i>	0.8 Gm.
Capsicum, in moderately fine powder, <i>three and two-tenths grammes.</i>	3.2 Gm.
Sugar of Milk, in fine powder, <i>six and one-half grammes.</i>	6.5 Gm.
Acacia, in fine powder, <i>one and six-tenths grammes.</i> ..	1.6 Gm.
Glycerin,	
Syrup, each, <i>a sufficient quantity,</i>	
To make <i>one hundred pills.</i> ..	100

Triturate the Resin of Podophyllum, Capsicum, Sugar of Milk, and Acacia together to produce a uniform powder. Incorporate with this the Extract of Belladonna Leaves and sufficient of a mixture of equal parts of Glycerin and Syrup to form a mass; divide it into *one hundred pills*.

Dose.—One pill.

PILULÆ RHEI COMPOSITÆ. Compound Pills of Rhubarb.

<i>Recipe.</i> —Rhubarb, in No. 60 powder, <i>thirteen grammes.</i>	13.0 Gm.
Purified Aloes, in fine powder, <i>ten grammes.</i>	10.0 Gm.
Myrrh, in fine powder, <i>six grammes.</i>	6.0 Gm.
Oil of Peppermint, <i>one-half cubic centimeter.</i>	0.5 Cc.
Water, <i>a sufficient quantity,</i>	
To make <i>one hundred pills.</i> ..	100

Mix the Oil of Peppermint intimately with the powders, then incorporate sufficient Water to form a mass; divide it into *one hundred pills*.

Dose.—Two pills.

Doses of Official Pills

As already mentioned, pills, troches, and suppositories differ from other classes of official pharmaceuticals taken internally in the fact that the finished products are subdivided into definite doses; hence while the dosage of other preparations are by weight or volume, we administer pills and troches by a certain number of subdivided particles. The pharmacopœial doses of all official pills are either one or two pills.

1 pill: Pills of opium; phosphorus; podophyllum, belladonna, and capsicum.

2 pills: Pills of aloes; aloes and iron; aloes and mastic; aloes and myrrh; asafetida; compound cathartic; vegetable cathartic; ferrous carbonate; ferrous iodide; compound laxative; compound rhubarb.

COMPRESSED TABLETS

These are lenticular masses of medicinal substances forced into a solid mass by compression. Compressed tablets were introduced by Brockedon in 1843, and soon became popular by reason of their convenience and simplicity of manufacture. It must be said, however, that compressed tablets are open to the possible objection of insolubility. Many substances, when compressed sufficiently firmly to hold together, are very difficultly soluble. In the manufacture of compressed tablets this should be borne in mind, and care should be

exercised in so blending the ingredients as to render them as soluble as possible under the circumstances.

The making of such compressed tablets depends chiefly on the proper blending of the ingredients, foreign substances being added either to secure solubility or to prevent sticking in the mold. These added inert constituents can be roughly grouped under the word excipient. For each combination of chemicals the excipient should be studied, even as we do in the case of pills, and the special directions are beyond the limits of this work. Suffice it here to cite a typical combination for a mass intended for compression. For this purpose a medicinal substance is combined with one-tenth its weight of sugar

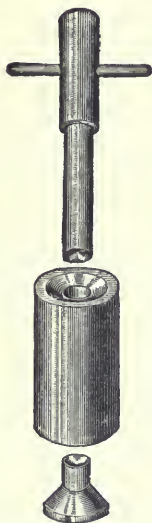


Fig. 192.—Compressed tablet mold.

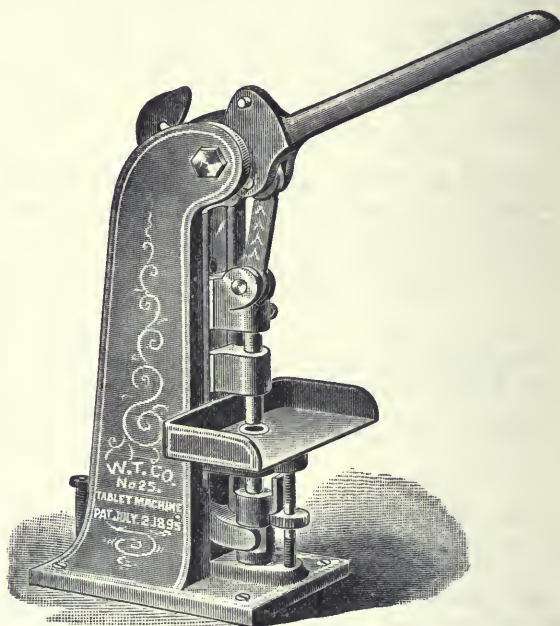


Fig. 193.—Compressed tablet machine.

and one-twentieth its weight of gum arabic. The mass is then mixed with a little water, granulated by passing through a coarse sieve, and then carefully dried. The dried granular mass is then sprayed with liquid petrolatum, ten or twelve drops being enough for a pound of mass. Such mass, on compression, yields a tablet that is fairly soluble, while the lubricant (liquid petrolatum) is squeezed out upon the surface of the compressed tablet, thus preventing the mass from sticking to the mold.

For the purpose of preventing the mass from adhering to the dies of the machine, French chalk is sometimes used, although in general practice liquid petrolatum is more satisfactory.

In making compressed tablets of quinine it is essential that the quinine be in a satisfactory condition before attempting to compress it, as otherwise they are almost sure to adhere to the mold. The

quinine should be at first sufficiently dried, and then should be granulated and sprayed with paraffin oil.

The apparatus used in compressing tablets consists, in its simplest form, of a steel cylinder pierced with a central bore, in which fits a plunger the end of which is convex, while at the other end of the cylinder fits the lower die, consisting of a cylindric piece of steel similar to the plunger, but shorter, terminating in a broad base. The cylinder is fitted to the lower die and the quantity of powder directed to be compressed is placed within the bore (Fig. 192). The plunger is then inserted, and the mass is then pressed against the lower die, forming a firm lenticular disk if the pressure be sufficient. The pressure is produced either by hitting the plunger with a hammer

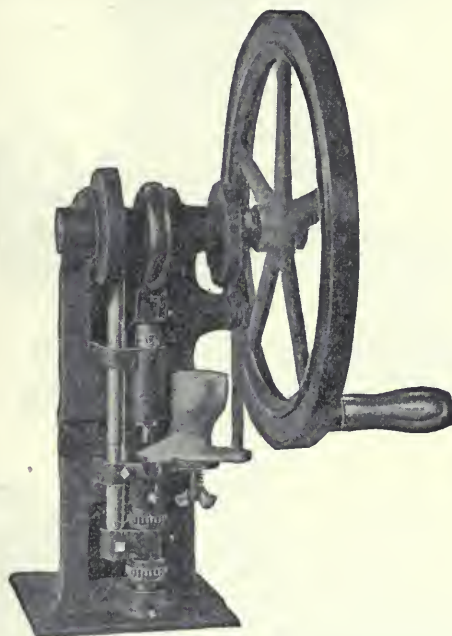


Fig. 194.—Stokes' "Eureka" tablet machine.

or mallet or by means of an iron lever. This lever is sometimes in the form of a long handle acting from a hinge. Of these machines, the one in which compression is performed with a hammer has fallen into disuse, on account of the noise accompanying the process and its general unsatisfactory character. The lever machine is simple and inexpensive, and should be found in every pharmacy for the manufacture of extemporaneous tablets. An excellent modification of the lever compressor is shown in Fig. 193. A very good investment for the prescription department is the more expensive machine, as shown in Fig. 194. In these machines the tablet mass is blended, then placed in the appropriate funnel, and the mechanism is worked by a crank on the end of the circular wheel which transmits power not merely to cause the automatic rise and fall of the two dies, thus pressing the tablets, but the same movement

also causes the throwing out of the funnel the quantity of the mass required for each tablet, and the flipping aside of the finished tablet. Such a tablet machine, known as the Stokes' "Eureka" machine, will readily make 100 tablets a minute. In large manufacturing concerns tablet machines having a higher efficiency are used, most notable of these being the Stokes'-Clark rotary tablet machine

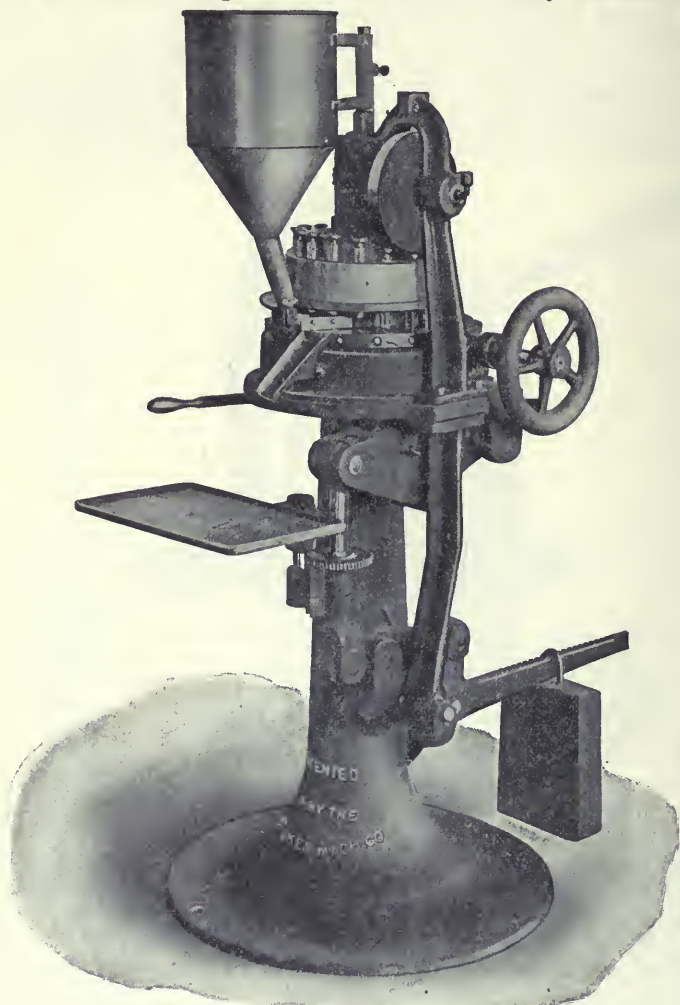


Fig. 195.—Stokes'-Clark rotary tablet machine.

(Fig. 195). In this machine there is a series of dies (12 to 25) operating on a circular plate, and being mechanically raised and lowered during the revolution of the plate by means of cams; each revolution of the plate, therefore, produces 12 to 25 tablets, and as the machine can be run very rapidly, it is capable of turning out from 30,000 to 50,000 tablets an hour.

TABLET TRITURATES

These are flat disks containing medicinal substances, prepared by making a paste of the substance and forcing them into the orifices of a special tablet triturating machine. This machine, as will be seen in Fig. 196, consists of a plate of steel or gutta-percha pierced with a number of accurately bored holes, all of which are of the same size.

The second part of the apparatus consists of a plate, which is studded with cylinders exactly fitting in the orifices of the perforated plate, and of sufficient length to project from $\frac{1}{8}$ to $\frac{1}{16}$ inch above the perforated plate when it is fitted on these cylinders.

In using such a tablet triturate machine, the mass, as above stated, is made into a thin paste by treatment with alcohol, and forced into the orifices of the perforated plate (which rests on a pill tile or glass plate) by rubbing with a spatula, and the mass is left in holes until firm. The plate is then placed over the studded plate and gradually pressed down over the projecting posts. As these posts pass into the orifices of the perforated plate the dry disks of medicinal substances rise upon the posts and finally emerge from the plate in which they were formed, and they are allowed to remain on the top

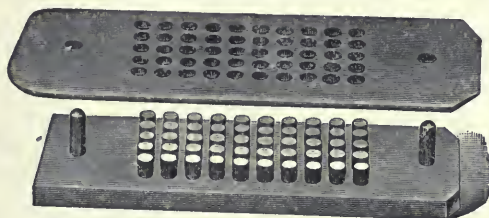


Fig. 196.—Tablet triturate mold.

of these posts until perfectly dried, when a gentle tilting of the apparatus, or tapping it with a spatula, will cause them to fall off.

Tablet triturates were introduced in order to make preparations similar to compressed tablets, but more soluble by reason of their lesser compression.

The disadvantage of these tablet triturates lies in the fact that when they become dry they are liable to disintegrate, and care must be taken in dispensing such in a pill box to provide a sufficient amount of cotton to prevent their shaking to and fro.

HYPODERMIC TABLETS

These are frequently prepared by the same method of making tablet triturates. They consist of medicinal substances (intended to be administered subcutaneously) blended with a substance known to be completely soluble and inert. For this purpose sodium sulphate has been used, but now sugar of milk, especially that from goat's milk, is preferred. Most of the hypodermic tablets are now made by compression.

TROCHES

Troches or lozenges are disk-like masses of medicinal substances intended to be administered by slowly dissolving in the mouth. By

reason of the method of administration the application of troches is limited to two classes of medicinal substances—first and usually, for the administration of medicines intended for application to the throat, and, secondly, in the administration of fairly mild and tasteless medicines in a palatable form. Since they are administered by slowly dissolving, no intensely bitter medicine should be used in the drug. Thus, to prepare troches of quinine sulphate would be scarcely short of senseless. A tasteless lozenge of quinine can, however, be made, and is described farther along.

Troches are manufactured in two ways—by massing and by compression. In making a lozenge by massing, the medicinal substances are combined with some mucilaginous substance, usually gum tragacanth, and moistened with sufficient water to make a mass, and are then worked up in a mortar similar to making a pill mass. When the mass is formed, it is rolled out with an ordinary biscuit roller on the biscuit board or pill tile, and from this rolled-out mass the lozenges are cut with an appropriate cutter. The details of the manufacture of such a mass will be found in the Appendix in the discussion of the manufacture of lozenges of potassium chlorate. The apparatus required, as mentioned above, is a biscuit board, which, however, in



Fig. 197.—Lozenge cutter.

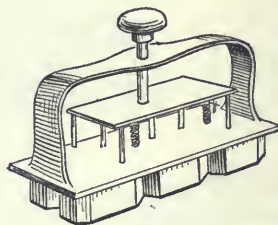


Fig. 198.—Sixfold lozenge cutter.

the writer's experience, is not as satisfactory, by reason of the warping of wood, as a glass plate, or, in making small quantities, a pill tile. For rolling out the mass any ordinary rolling-pin will answer, the size depending upon the number of troches intended to be made. As to the lozenge cutters, they are generally hollow cylinders of tin tipped with steel, as shown in Fig. 197. The shape of this can be either round or hexagonal. Special forms of lozenge cutters for cutting six at a time have been devised (see Fig. 198).

The making of lozenges by compression is similar to making compressed tablets, the only difference being in the size of the mold. In the case of compressed tablets, the die arranged for 1-grain masses has a diameter of $\frac{1}{4}$ inch; while the 5-grain die is $\frac{3}{8}$ inch in diameter. The lozenge being usually larger, a special die is provided, with all compressed tablet machines, in which the diameter is $\frac{1}{2}$ to $\frac{3}{4}$ inch. Such a die is attached to the tablet machine, the powdered constituents blended in the proper way, and compressed either by hand or by machine.

For throat troubles the compressed lozenges are as valuable as

the massed lozenges, because in such cases the medicinal ingredients of these lozenges act best when they are slowly dissolved.

BACILLI

These are special forms of lozenges, consisting of cylinders about an inch in length and $\frac{1}{8}$ to $\frac{1}{4}$ inch in diameter. The best illustration of bacilli are the black licorice lozenges so largely sold in America these days. Such bacilli are made by taking the mass and either rolling it into a cylinder by hand and dividing on a pill-cutting machine, or, better still, forcing it through a bougie machine (p. 338), from which it emerges as a narrow cylinder which is cut by a knife into short pieces.

TABLE OF OFFICIAL LOZENGES

(ALL GALENIC)

<i>Process.</i>	<i>Uses.</i>	<i>Excipient.</i>	<i>Preparations.</i>
All by massing and cutting.	Throat lozenges.	Tragacanth and stronger orange flower water.	Troches of tannic acid.
		Tragacanth and syrup of tolu.	Troches of gambir.
		Tragacanth and water.	Troches of krameria.
			Troches of ammonium chloride.
		Acacia and syrup of tolu.	Troches of potassium chlorate.
		Acacia and water.	Troches of cubeb.
	Not for throat.	Tragacanth and stronger orange flower water.	Troches of glycyrrhiza and opium.
		Mucilage of tragacanth.	Troches of santolin.
			Troches of sodium bicarbonate.

SPECIAL NOTES ON OFFICIAL LOZENGES

TROCHISCI ACIDI TANNICI. Troches of Tannic Acid.

<i>Recipe.</i> —Tannic Acid, six grammes.....	6 Gm.
Sugar, in fine powder, sixty-five grammes.....	65 Gm.
Tragacanth, in fine powder, two grammes.....	2 Gm.
Stronger Orange Flower Water, a sufficient quantity,	

To make one hundred troches. . 100

Rub the powders together until they are thoroughly mixed; then, with Stronger Orange Flower Water, form a mass, to be divided into one hundred troches.

TROCHISCI AMMONII CHLORIDI. Troches of Ammonium Chloride.

<i>Recipe.</i> —Ammonium Chloride, in fine powder, 10 grammes.....	10 Gm.
Extract of Glycyrrhiza, in fine powder, twenty grammes.....	20 Gm.
Tragacanth, in fine powder, two grammes.....	2 Gm.
Sugar, in fine powder, forty grammes.....	40 Gm.
Syrup of Tolu, a sufficient quantity,	

To make one hundred troches. . 100

Rub the powders together until they are thoroughly mixed; then, with Syrup of Tolu, form a mass, to be divided into one hundred troches.

TROCHISCI CUBEÆ. Troches of Cubeb.

<i>Recipe.</i> —Oleoresin of Cubeb, two grammes.....	2 Gm.
Oil of Sassafras, one cubic centimeter.....	1 Cc.
Extract of Glycyrrhiza, in fine powder, twenty-five grammes.....	25 Gm.
Acacia, in fine powder, twelve grammes.....	12 Gm.
Syrup of Tolu, a sufficient quantity,	
To make one hundred troches..	100

Rub the powders together until they are thoroughly mixed; then add the Oleoresin and the Oil, and incorporate them with the mixture. Lastly, with Syrup of Tolu, form a mass, to be divided into *one hundred troches*.

TROCHISCI GAMBIR. Troches of Gambir.

<i>Recipe.</i> —Gambir, in fine powder, six grammes.....	6 Gm.
Sugar, in fine powder, sixty-five grammes.....	65 Gm.
Tragacanth, in fine powder, two grammes.....	2 Gm.
Stronger Orange Flower Water, a sufficient quantity,	
To make one hundred troches..	100

Rub the powders together until they are thoroughly mixed; then, with Stronger Orange Flower Water, form a mass, to be divided into *one hundred troches*.

Remarks.—These lozenges replace the troches of catechu which were official in the pharmacopœia of 1890. It has already been mentioned (p. 227) that gambir (pale catechu) was made official at the last revision instead of catechu, and likewise all former official preparations of catechu have been replaced by similar preparations of gambir.

TROCHISCI GLYCYRRHIZÆ ET OPII. Troches of Glycyrrhiza and Opium.

<i>Recipe.</i> —Extract of Glycyrrhiza, in fine powder, fifteen grammes.....	15.0 Gm.
Powdered Opium, one-half gramme.....	0.5 Gm.
Acacia, in fine powder, twelve grammes.....	12.0 Gm.
Sugar, in fine powder, twenty grammes.....	20.0 Gm.
Oil of Anise, two-tenths of a cubic centimeter.....	0.2 Cc.
Water, a sufficient quantity,	
To make one hundred troches..	100

Rub the powders together until they are thoroughly mixed; then, add the Oil of Anise (equivalent to about 4 drops), and incorporate it with the mixture. Lastly, with Water, form a mass, to be divided into *one hundred troches*.

Remarks.—These lozenges enjoy considerable popularity under the name of *Wistar's throat lozenges*. They and troches of cubeb are the smallest official lozenges, weighing less than $\frac{1}{2}$ gramme. For this reason they are usually dispensed in the form of bacilli.

TROCHISCI KRAMERIÆ. Troches of Krameria.

<i>Recipe.</i> —Extract of Krameria, six grammes.....	6 Gm.
Sugar, in fine powder, sixty-five grammes.....	65 Gm.
Tragacanth, in fine powder, two grammes.....	2 Gm.
Stronger Orange Flower Water, a sufficient quantity,	
To make one hundred troches..	100

Rub the powders together until they are thoroughly mixed; then, with Stronger Orange Flower Water, form a mass, to be divided into *one hundred troches*.

TROCHISCI POTASSII CHLORATIS. Troches of Potassium Chlorate.

Recipe and full details of manipulation will be found in Part VII.

Remarks.—In passing, it may be stated that the potassium chlorate lozenges of the present pharmacopœia are exactly one-half the size of those recognized by the pharmacopœia of 1890, and that they must not be confounded with the compressed *tablets of the potassium chlorate*, which usually consists of five grains of that chemical in compressed form.

In making the troches, great care should be taken in mixing the potassium chlorate with the sugar, that combination, as mentioned on p. 442, being apt to explode. Not only is caution required in the manufacture, but also it is well to warn the purchaser that these lozenges should not be subjected to any violent concussion, as such is apt to produce a dangerous explosion.

TROCHISCI SANTONINI. Troches of Santonin.

<i>Recipe.</i> —Santonin, in fine powder, <i>three grammes</i>	3 Gm.
Sugar, in fine powder, <i>ninety grammes</i>	90 Gm.
Tragacanth, in fine powder, <i>three grammes</i>	3 Gm.
Stronger Orange Flower Water, <i>a sufficient quantity</i> ,	

To make one hundred troches.. 100

Rub the powders together until they are thoroughly mixed; then, with Stronger Orange Flower Water, form a mass, to be divided into *one hundred troches*.

Troches of Santonin should be kept in dark amber-colored vials.

Remarks.—These lozenges are popular as *worm candy*, because easily administered to children. Each contains about one-half grain of santonin.

Santonin is prone to decomposition when exposed to light; hence the pharmacopœia directs that the lozenges of santonin should be kept in dark-colored bottles.

TROCHISCI SODII BICARBONATIS. Troches of Sodium Bicarbonate.

<i>Recipe.</i> —Sodium Bicarbonate, <i>eighteen grammes</i>	18 Gm.
Sugar, in fine powder, <i>fifty-four grammes</i>	54 Gm.
Myristica, bruised, <i>one gramme</i>	1 Gm.
Mucilage of Tragacanth, <i>a sufficient quantity</i> ,	

To make one hundred troches.. 100

Triturate the Myristica with the Sugar, gradually added, until they are reduced to a fine powder, and mix this intimately with the Sodium Bicarbonate; then, with the Mucilage of Tragacanth, form a mass, to be divided into *one hundred troches*.

Remarks.—These lozenges are given as an antacid in cases of flatulence.

A few unofficial forms of lozenges are worthy of notice.

Jujube paste lozenges formerly enjoyed large popularity. The original jujube paste was the mucilaginous exudation from *Zizyphus sativa*, but it has long since been replaced by a base consisting of acacia, 18; sugar, 6; water, 80; evaporate in water-bath to 30 parts.

With this base various medicines can be incorporated, and lozenges so prepared have been highly recommended by throat surgeons.

A lozenge of "tasteless quinine" is largely used in the Southern States for administering this valuable drug to children. The quinine salt selected as the least bitter is the tannate, and 1 grain of this salt, incorporated with chocolate, 5 grains, confectioners' sugar, 10 grains, and worked into a mass with mucilage of tragacanth and molded into a lozenge, furnishes the needed pharmaceutic.

Doses of Official Troches

No doses are assigned troches by the pharmacopœia, it being assumed that they can be administered *ad libitum*. Warning must be given, however, that indiscriminate consumption of official lozenges may lead to grave results. Especially is this true with Wistar's lozenge, which contains opium; troches of potassium chlorate, the irritating effect of this chemical being mentioned on p. 442; and troches of santonin, of which one is a sufficient dose for a child.

CHAPTER XX

SUPPOSITORIES

THE present pharmacopœia discusses this important class of pharmaceuticals in general directions that are so clear, so sensible, and so thorough that their reproduction on these pages furnishes *almost* all that need be said on the subject; hence we will turn forthwith to these general directions.

SUPPOSITORIA. Suppositories.

Suppositories are solid bodies of various weights and shapes, adapted for introduction into the different orifices of the human body, and melting readily at blood heat. The vehicles usually employed are Oil of Theobroma, Glycerinated Gelatin, or Sodium Stearate.

For suppositories made with Oil of Theobroma the following general processes may be employed:

Recipe.—**The Medicinal Substance, the prescribed quantity.**
Oil of Theobroma, grated, a sufficient quantity.

Reduce the Medicinal Substance, if dry, to a very fine powder, or, if an extract, soften it with an appropriate liquid, then mix it thoroughly in a mortar with about an equal weight of grated Oil of Theobroma, and incorporate the remainder of the Oil of Theobroma until a homogeneous, plastic mass is obtained, adding, if necessary, a small quantity of Expressed Oil of Almond. Roll the mass on a graduated tile until a cylinder of the proper length is formed, divide this into the required number of equal parts, and with a spatula, or other convenient mechanical aid, form them into the desired shape.

If the process of fusion is preferred, mix the Medicinal Substance with about an equal weight of grated Oil of Theobroma, as above directed, then thoroughly incorporate it with the remainder of the Oil of Theobroma, previously melted by a gentle heat, in a suitable vessel provided with a lip; then allow it to cool to about 38° C. (100.4° F.), and, when the mixture begins to congeal, pour it immediately

into suitable well-cooled molds. Keep the molds at a freezing temperature until the suppositories have hardened and are ready to be removed.

For suppositories containing chloral, phenol, their derivatives, or substances which soften the vehicle, raise the melting-point of the Oil of Theobroma by the addition of from 10 to 15 per cent. of spermaceti, but the melting-point must not be raised above 37° C. (98.6° F.).

For suppositories made with Glycerinated Gelatin the following process may be used:

Recipe.—The Medicinal Substance, the prescribed quantity.

Glycerinated Gelatin,

Glycerin,

Water, each, a sufficient quantity.

Mix the Medicinal Substance, if solid and soluble, in Water or Glycerin, or if a miscible liquid, with a little Water, and add sufficient Glycerin to make the weight of the mixture one-half that of the finished mass. Then thoroughly incorporate it with an equal weight of melted Glycerinated Gelatin, and pour it at once into suitable molds which have been greased with a small quantity of petrolatum. Cool the molds thoroughly before removing the suppositories. Molds for urethral suppositories should be warmed sufficiently before pouring the mass to facilitate the proper filling of the mold. Suppositories having a firmer consistence may be prepared by substituting Mucilage of Acacia for a portion of the Water or Glycerin.

If the Medicinal Substance be insoluble in Water or Glycerin, thoroughly levigate it in a warm mortar with a sufficient quantity of Glycerin to make the weight of the mixture one-half that of the finished mass. Then thoroughly incorporate it with an equal weight of melted Glycerinated Gelatin, and pour it into suitable molds, as above directed. With bulky powders about one-half of the Glycerin may be replaced with Water before levigation. Glycerinated Gelatin suppositories should be protected against the effects of heat and moisture and dry air by keeping them in tightly closed containers in a cool place.

Rectal Suppositories should be cone shaped or spindle shaped, and when made from Oil of Theobroma should weigh about *two grammes*.

Urethral Suppositories (Bougies) should be pencil shaped, pointed at one extremity, and either *seven centimeters* in length, weighing about *two grammes*, or *fourteen centimeters* in length, weighing about *four grammes*, when made with Glycerinated Gelatin. If prepared with Oil of Theobroma they should weigh about one-half the above quantities.

Vaginal Suppositories should be globular or oviform in shape and weigh about *ten grammes* if made with Glycerinated Gelatin, and about *four grammes* if made with Oil of Theobroma.

Remarks.—From the pharmacopœial directions just given, it will be noticed that suppositories are administered by inserting into rectum, vagina, or urethra, and that, according to place of insertion and material from which made, they are of different sizes and shapes. This data can be tabulated as follows:

OFFICIAL SUPPOSITORIES

<i>Application.</i>	<i>Composition.</i>	<i>Shape.</i>	<i>Weight.</i>
Rectal.	Oil of theobroma.	Cone or spindle shaped.	2 grammes.
Urethral.	Glycerinated gelatin.	Pencil shaped.	2 to 4 grammes.
	Oil of theobroma.	Pencil shaped.	1 to 2 “
Vaginal.	Glycerinated gelatin.	Globular or oviform.	10 grammes.
	Oil of theobroma.	Globular or oviform.	4 grammes.

The ideal suppository consists of medicine blended with some inert base which will not liquefy at ordinary temperatures, but will melt at the temperature of the human body. The best substance adapted

for this purpose is oil of theobroma (butter of cacao); that is, the official solid fatty oil, which, as is explained on p. 730, is a hard, firm mass, which, however, melts to an oily liquid at 90° to 95° F. In America practically all suppositories are made from this substance, its use having been suggested by the American, A. B. Taylor, in 1852. In England glycerinated gelatin is popular as a suppository base, but this base possesses the decided disadvantage that the choice of medicinal ingredients to be blended with it is somewhat limited. Thus, under no circumstances should tannin or tannin-bearing drugs be combined with gelatin, for if this is done, an insoluble tannate of gelatin (leather) is formed.

A third suppository base, used only in one case, and that an official illustration, is the solidification of glycerin by means of stearic acid. This will be discussed in considering the official glycerin suppositories.

Manufacture of Suppositories.—There are three important ways of making suppositories—by *rolling*, by *molding*, and by *compressing*.

Of these processes, manufacture by rolling, that is, making into a mass, rolling out, cutting cylinder, and finally shaping by hand, and also by molding (or fusion), is outlined in above general directions, while minutiae of each process, applied in special cases, is given in Part VII.

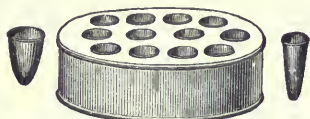


Fig. 199.—Individual suppository molds.

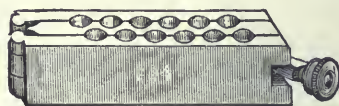


Fig. 200.—Divided suppository mold.

At this place it is, therefore, only necessary to say a few words concerning the apparatus (molds) used in preparing suppositories by fusion.

These molds are made of brass, usually nickel plated, and, as mentioned above, before using are to be thoroughly chilled before the suppository mass is placed therein.

The first form of the suppository mold was the individual mold, consisting of a conic piece of steel hollowed out to the shape of a suppository, and a set of these molds is fitted into an appropriate holder which can then be immersed in ice-water, or a mixture of ice-water and salt, as shown in Fig. 199. These molds are absolutely useless in the modern pharmacy, there being no way of opening them to remove a possibly refractory suppository; and, moreover, the quantity of metal in the mold is not sufficient to insure a thoroughly uniform chilling. If such is used, the one way of removing the suppository is by tapping the mold, and if the mass sticks to the mold, nothing can be done except by digging it out by means of a wire or a file.

Divided molds consist of larger pieces of steel containing orifices for 6, 12, 24, or even a larger number of suppositories, so that they can be taken apart, thus dividing the mold in two pieces, as shown in Fig. 200. While being used, the two pieces are held together by means of clamps or pegs. Such molds are the most satisfactory

that can be used, and if they have been left on the ice long enough to insure thorough chilling, the manufacture of suppositories with such molds yields most satisfactory results.

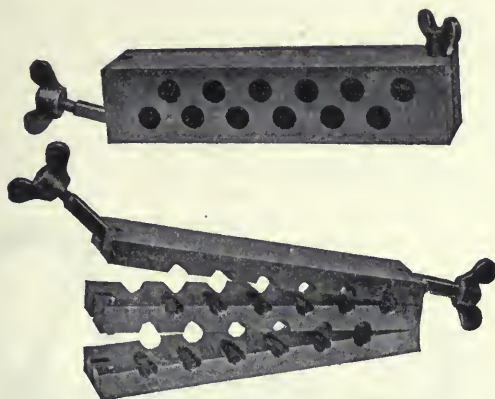


Fig. 201.—Benton-Hall suppository mold.

Quite a number of makes of divided suppository molds are on the market, and none of them is better than the hinged Benton-Hall mold, as shown in Fig. 201. It will be seen from the figure that this

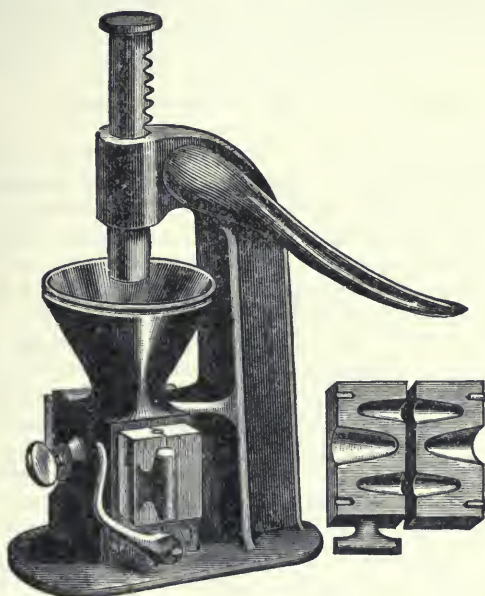


Fig. 202.—Suppository compressor.

is on the same principle as the divided molds just described, but has the advantage of having a set of orifices at both top and bottom.

Suppositories by compression are made by mixing the medicinal substance with finely grated oil of theobroma and compressing it into the mold by means of appropriate leverage. Several varieties of such compressing machines are on the market. Earliest of these was the Archibald suppository machine (shown in Fig. 202), in which the mixture was passed through a funnel in which works a steel plunger, which forces the mass into a mold placed underneath the funnel. This form of suppository machine is not highly satisfactory, however, and is rarely used in these days.

The Whitall-Tatum suppository machine works far more efficiently. The cylinder is smaller and rests horizontally on the table rather than vertically (Fig. 203).

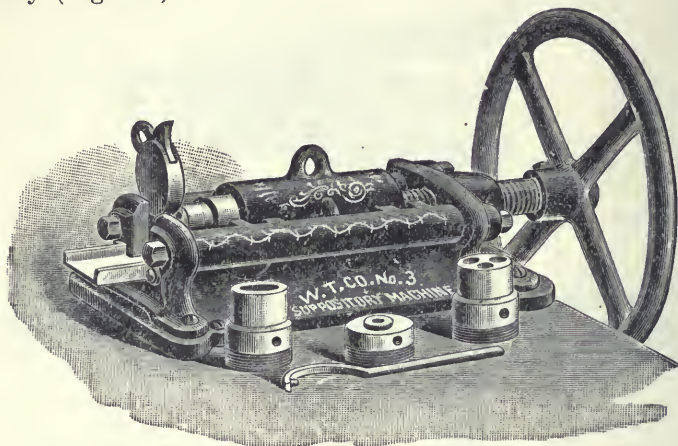


Fig. 203.—Whitall-Tatum suppository machine.

In making *bougies* by compression instead of the cone-shaped mold the mass is pressed through a die having a single but perfectly true cylindric orifice bored through it. The pressure of the plunger within the cylinder forces the mass through this narrow orifice, with the result of producing long, worm-like masses.

In lieu of compressing machines bougies can be made by working up the mass by hand and rolling it out as one would a pill cylinder. Or, if it is preferred to make the urethral suppository by fusion, the fused mass can be sucked up into a glass tube, which has been previously oiled. It will be noticed from the official directions given above that compressed suppositories have not been given official sanction.

The pharmacopœia recognizes but one suppository by name.

SUPPOSITORIA GLYCERINI. Suppositories of Glycerin.

Recipe and details of manufacture of this important preparation are left for practice work in Part VII.

Remarks.—The mass from which the suppositories are made is prepared by treating glycerin with sodium carbonate and stearic acid, thus forming a glyceryl-sodio stearate, the chemistry of which will be discussed in Part VII.

It is here necessary merely to call attention to the fact that a very small quantity (less than 10 per cent.) of solids are able to solidify glycerin.

In place of ordinary suppositories there have been placed on the market the so-called *suppository capsules*. They consist either of a hollow mass of oil of theobroma, provided with a plug of the same material used to close the capsule; while others are made of gelatin, similar to gelatin capsules, only larger. In both cases the medicine intended to be administered as a suppository is supposed to be introduced into these capsules, which are then sealed and are then ready for insertion. It is hardly necessary to say that the careful pharmacist will avoid dispensing such suppositories. In the first place, one of the objects of using oil of theobroma mass is that the active medicinal ingredients shall be properly diluted, and to place these ingredients in such capsules, even though worked up with a small quantity of oil of theobroma, does not yield a suppository of a value equal to the one made by mixing the medicaments with the diluents.

CHAPTER XXI

SOLID PREPARATIONS FOR EXTERNAL USE (CERATES, OINTMENTS, CATAPLASMS, PLASTERS, AND PAPERS)

CERATES

THESE are combinations of medicinal substances with fats and waxes intended to be used by spreading on cloth or paper, applying as one would a plaster. As the name suggests, cerates (from the Latin *cera*, wax) are supposed to contain wax, and a successful cerate should be soft enough to spread on cloth or paper, and yet thick enough not to become thin and oily in hot weather.

The statement as to the origin of the word cerate has lost some of its force since the last pharmacopœial revision, as one cerate now official (cerate of lead subacetate) contains no wax, paraffin being the hardening agent.

The fats used in the manufacture of cerates run the gamut of all available fatty substances. Lard is generally employed, but sometimes fatty oils are used; while in the present pharmacopœia petrolatum and lanolin are prescribed in several cases. According to the amount of semisolid fat, such as petrolatum or lard, or of oil employed, the amount of wax required must be varied; oils, of course, requiring a larger amount. Again, in several recipes for cerates and ointments in the present pharmacopœia directions are given to use more wax in hot weather than during the cold weather.

The official cerates are made either by *fusion* or by *incorporation*. In the manufacture by fusion the constituents are melted together, the melted mass strained through muslin into a mortar, and triturated until completely solidified. In incorporation the medicinal substance

is placed on an ointment slab or in the mortar, and mixed with the fatty ingredients; in the first case, by rubbing with the spatula, and in the second, by trituration with the pestle.

In the present pharmacopœia all official cerates are made by fusion, the one which the pharmacopœia of 1890 directed, prepared by incorporation (cerate of lead subacetate), has no longer camphor cerate as its base, but is made with melted mixture of wool-fat, white petrolatum, and paraffin.

TABLE OF OFFICIAL CERATES
(ALL GALENIC AND ALL MADE BY FUSION)

Base.	Preparations.
White wax, white petrolatum, and benzoinated lard.	Cerate.
Wool-fat, paraffin, and white petrolatum.	Camphor cerate.
Yellow wax and lard.	Cerate of lead subacetate.
Yellow wax, prepared suet, and linseed oil.	Rosin cerate.
Yellow wax, lard, and liquid petrolatum.	Rosin cerate, compound.
	Cantharides cerate.

SPECIAL NOTES ON OFFICIAL CERATES

CERATUM. Cerate.

<i>Recipe.</i> —White Wax, three hundred grammes.....	300 Gm.
White Petrolatum, two hundred grammes.....	200 Gm.
Benzoinated Lard, five hundred grammes.....	500 Gm.
To make one thousand grammes..	1000 Gm.

Melt the White Wax, add the White Petrolatum, then the Benzoinated Lard, continuing the heat until the mixture is liquefied, and stir it constantly until it congeals.

For use in southern latitudes, and during the heated season in other localities, fifty grammes of Benzoinated Lard may be replaced by an equal quantity of White Wax.

Remarks.—This preparation, commonly called *simple cerate*, is made by the fusion of 30 per cent. white wax, 20 per cent. white petrolatum, and 50 per cent. benzoinated lard. The recipe given by the present pharmacopœia for this and other cerates differs from those of the pharmacopœia of 1890 by the use of petrolatum products, and, in one case, of wool-fat. Thus the old recipe for cerate called for 30 per cent. white wax and 70 per cent. lard.

Simple cerate is rarely used except in combination with medicinal substance.

CERATUM CAMPHORÆ. Camphor Cerate.

<i>Recipe.</i> —Camphor Liniment, one hundred grammes.....	100 Gm.
White Wax, three hundred and fifty grammes.....	350 Gm.
White Petrolatum, one hundred and fifty grammes.....	150 Gm.
Benzoinated Lard, four hundred grammes.....	400 Gm.
To make one thousand grammes..	1000 Gm.

Melt the White Wax and the White Petrolatum, then the Benzoinated Lard, and continue the heat until the mixture is liquefied. While the mixture is cooling, add the Camphor Liniment, and incorporate thoroughly by stirring until it congeals.

Remarks.—Made by combining camphorated oil (camphor liniment) with a melted mixture of white wax, white petrolatum, and

benzoinated lard. This is not the French "*pomade camphree*," used largely in southern States, and found in the National Formulary under the title "*unguentum camphoræ*." The latter contains 20 per cent. camphor, 10 per cent. white wax, and 60 per cent. lard; hence is many times stronger than the official cerate, which contains 10 per cent. of camphor liniment, while the liniment contains 20 per cent. camphor. Hence the cerate contains but 2 per cent. camphor.

CERATUM CANTHARIDIS. Cantharides Cerate.

<i>Recipe.</i> —Cantharides, in No. 60 powder, three hundred and twenty grammes.....	320 Gm.
Liquid Petrolatum, one hundred and fifty grammes.....	150 Gm.
Yellow Wax, one hundred and eighty grammes.....	180 Gm.
Rosin, one hundred and eighty grammes.....	180 Gm.
Lard, one hundred and seventy grammes.....	170 Gm.
To make one thousand grammes..	1000 Gm.

Mix the Cantharides with the Liquid Petrolatum, and set the mixture aside, well covered, in a warm place, for forty-eight hours. Then add it to the Rosin, Yellow Wax, and Lard, previously melted and strained through muslin, and keep the mixture in a liquid condition by means of a water-bath, stirring occasionally, for one hour. Finally, remove it from the bath, and stir the mixture until it begins to congeal.

Remarks.—This is prepared by macerating powdered cantharides with liquid petrolatum and mixing the other fatty constituents previously brought to fusion, keeping the mixture in a liquid condition for an hour. After the heat is removed the mass is stirred until cooled. The cantharides cerate is used for spreading what is commonly known as *fly-blister*, this being the cerate spread on muslin or adhesive plaster in a layer about $\frac{1}{4}$ inch thick. The spreading of the fly-blister is similar to the spreading of plasters, a piece of the adhesive plaster of the appropriate size being cut off from the roll, the area to be covered inclosed by strips of stiff paper, and the cerate spread by means of a spatula over the exposed portion of the plaster. When spread, the plaster is finished by sprinkling with a small quantity of vinegar of cantharides or a minute trace of cantharides. Cantharides cerate is the most satisfactory method of producing a blister, and in inflammatory diseases the fly-blister is of the utmost value.

CERATUM PLUMBI SUBACETATIS. Cerate of Lead Subacetate.

<i>Recipe.</i> —Solution of Lead Subacetate, twenty grammes....	20 Gm.
Wool-fat, twenty grammes.....	20 Gm.
Paraffin, twenty grammes.....	20 Gm.
White Petrolatum, thirty-eight grammes.....	38 Gm.
Camphor, two grammes.....	2 Gm.
To make one hundred grammes..	100 Gm.

To the Melted Wool-fat in a warm mortar gradually add the Solution of Lead Subacetate, and incorporate it by slow trituration. To the mixture add the White Petrolatum and Paraffin, previously melted, and in which the Camphor has been dissolved; mix thoroughly until homogeneous.

Remarks.—This preparation, known as *Goulard's cerate*, is made by combining a 20 per cent. solution of lead subacetate (Goulard's extract) with melted wool-fat, and adding to this a melted mixture

of white petrolatum and paraffin containing 2 per cent. camphor. The use of paraffin and petrolatum in this recipe is an advantage over the lard and wax used in the recipe of 1890, as the cerate made by the latter process assumed, on standing, a pink tint and became rancid.

CERATUM RESINÆ. Rosin Cerate.

<i>Recipe.</i> —Rosin, three hundred and fifty grammes.....	350 Gm.
Yellow Wax, one hundred and fifty grammes.....	150 Gm.
Lard, five hundred grammes.....	500 Gm.
To make one thousand grammes..	1000 Gm.

Melt the Rosin, add the Yellow Wax and the Lard, and continue the heat until liquefied, then strain the liquid through muslin, and allow it to congeal with occasional stirring.

In cold weather five hundred and thirty grammes of Lard and one hundred and twenty grammes of Yellow Wax may be used.

Remarks.—This is best known by its synonym, *basilicon ointment*. It is made by the fusion of rosin, yellow wax, and lard, and it is worth noting that the pharmacopœia directs the changing of the proportion of the constituents according as the cerate is made in hot or cold weather.

The cerate has great popularity as a “drawing salve” for bringing boils “to a head.”

CERATUM RESINÆ COMPOSITUM. Compound Rosin Cerate.

<i>Recipe.</i> —Rosin, two hundred and twenty-five grammes.....	225 Gm.
Yellow Wax, two hundred and twenty-five grammes.....	225 Gm.
Prepared Suet, three hundred grammes.....	300 Gm.
Turpentine, one hundred and fifteen grammes.....	115 Gm.
Linseed Oil, one hundred and thirty-five grammes..	135 Gm.
To make one thousand grammes..	1000 Gm.

Melt the Rosin, Yellow Wax, Turpentine, and Prepared Suet; to this add the Linseed Oil, and continue the heat until the mixture is liquefied; then strain it through coarse muslin, and stir it until it begins to congeal.

Remarks.—This official—re-introduced into the pharmacopœia after thirty years of non-recognition by our official standard—is largely used in certain sections of the country as *Deshler's salve*.

OINTMENTS

Ointments are semisolid preparations containing medicinal substance, blended with fatty matter of sufficient softness to permit their being applied to the skin by inunction. Ointments are always of softer consistence than are cerates, and have a lower melting-point, less wax being used. Thus while *cerate* of the present pharmacopœia contains 30 per cent. white wax, the official ointment contains but 20 per cent. of that hardening agent.

Ointments are manufactured either by *fusion* or by *incorporation*. In fusion the fatty substances are liquefied by the aid of gentle heat, and the medicinal substances are incorporated either while the fats are liquefied or after solidification. In the case of the ointments

made by incorporation the medicinal substances are rubbed up with solid fatty matter, either in a mortar or on an ointment slab. When pestle and mortar are used for the purpose of incorporation it is advisable to have the latter of a special flat shape, as shown in Fig. 204. An ointment prepared by fusion is usually finished by passing the fused mass through the strainer into a mortar, and tritulating the mass until solidified. This has the effect of not only making the ointment smoother, but also lighter in color. In some of the ointments "whipping" is important in order to obtain a handsome product. Thus, in making cold-cream, the finished product is made light and fluffy by beating the mass with an egg-beater, and in making large quantities by working up in an emulsion machine (p. 268).

When an ointment is prepared by incorporation on a slab, the apparatus needed consists of an ointment slab and a spatula. For the slab the ordinary pill tile can be used, although more handsome forms of slabs, made of plate glass, are obtainable (Fig. 205). Some years since, the use of pads of parchment paper was suggested instead of the slab, the idea being that, after the finishing of each ointment, the sheet of paper on which the ointment was worked up would be



Fig. 204.—Ointment mortar.

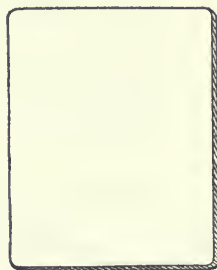


Fig. 205.—Ointment slab.

torn off and thrown away. This scheme, however, is not very satisfactory, inasmuch as in many cases it is advisable to triturate the chemicals incorporated in the ointment with considerable pressure, and the excuse for the use of the paper pad—saving the trouble of cleaning the ointment slab—was scarcely worth notice.

The spatulas used in incorporating ointments should be long and pliable, those made of nickel-plated steel being preferable. The iron spatula, however, can be used in all cases with those chemicals which will not attack iron, but must not be used in making citrine ointment (containing nitric acid) nor iodine ointment. For the latter purpose the steel spatula, coated with gutta-percha or rubber, is very well adapted, although the objection to such gutta-percha spatulas is their tendency to peel. In preparing the ointment by incorporation on the slab the chemicals, which should be in a very fine divided form, are further subdivided by the process of levigation, using the end of the spatula blade in place of the muller, and a portion of the ointment base as the lubricator. In the making of the ointment of the red mercuric oxide the chemical is to be levigated with water with the gutta-percha spatula—in this case it being essential that the chemical be reduced to the finest possible form of powder.

TABLE OF OFFICIAL OINTMENTS

(ALL GALENIC EXCEPT OINTMENT OF MERCURIC NITRATE).

<i>Process.</i>	<i>Base.</i>	<i>Preparations.</i>
Fusion.	Lard.	Ointment of mercuric nitrate.
	Benzoinated lard.	Ointment of chrysarobin.
		Ointment of zinc oxide.
	Lard and white wax.	Ointment.
	Lard and yellow wax.	Tar ointment.
	White petrolatum.	Ointment of phenol.
		Ointment of zinc stearate.
	White petrolatum and paraffin.	Ointment of boric acid.
	Expressed oil of almond, spermaceti, and white wax.	Ointment of rose water.
	Olive oil.	Diachylon ointment.
Incorporation.	Lard.	Iodoform ointment.
	Benzoinated lard.	Iodine ointment.
		Ointment of potassium iodide.
		Sulphur ointment.
		Veratrine ointment.
	Benzoinated lard and prepared suet.	Mercurial ointment.
	Benzoinated lard and hydrous wool-fat.	Belladonna ointment.
	Ointment (U. S. P.).	Stramonium ointment.
		Ointment of nutgall.
		Ointment of tannic acid.
	Petrolatum.	Blue ointment.
	Petrolatum and hydrous wool-fat.	Ointment of yellow mercuric oxide.
		Ointment of red mercuric oxide.
	White petrolatum and hydrous wool-fat.	Ointment of ammoniated mercury.

SPECIAL NOTES ON OFFICIAL OINTMENTS

UNGUENTUM. Ointment.

<i>Recipe.</i> —White Wax, two hundred grammes.....	200 Gm.
Benzoinated Lard, eight hundred grammes.....	800 Gm.
To make one thousand grammes..	1000 Gm.

Melt the White Wax, add the Benzoinated Lard, and heat gently until liquefied ; then stir the mixture until it congeals.

Remarks.—This preparation, commonly called *simple ointment*, is used almost entirely as a base for medicated ointments. As seen from above table, it is so employed in two of the ointments of the present pharmacopœia; while 4 of the 23 ointments recognized in the pharmacopœia of 1890 used the same base.

UNGUENTUM ACIDI BORICI. Ointment of Boric Acid.

<i>Recipe.</i> —Boric Acid, in fine powder, one hundred grammes..	100 Gm.
Paraffin, one hundred grammes.....	100 Gm.
White Petrolatum, eight hundred grammes.....	800 Gm.
To make one thousand grammes..	1000 Gm.

Melt the Paraffin, add the White Petrolatum, and heat gently for ten minutes. Then gradually add the hot liquid to the Boric Acid, contained in a warm mortar, triturating thoroughly, and stir the mixture until it congeals.

Remarks.—This new official contains 10 per cent. of boric acid in an ointment base consisting of paraffin and white petrolatum.

UNGUENTUM ACIDI TANNICI. Ointment of Tannic Acid.

<i>Recipe.</i> —Tannic Acid, twenty grammes.....	20 Gm.
Glycerin, twenty grammes.....	20 Gm.
Ointment, sixty grammes.....	60 Gm.
To make one hundred grammes..	100 Gm.

Dissolve the Tannic Acid in the Glycerin, with the aid of a gentle heat, and mix the solution thoroughly with the Ointment in a mortar, avoiding the use of iron utensils.

Remarks.—This ointment is made by combining a glycerinic solution of tannin with simple ointment. Iron vessels must be avoided in its manufacture, since tannate of iron would form and the product would blacken.

UNGUENTUM AQUÆ ROSÆ. Ointment of Rose Water.

Recipe and details of manipulation will be found in Part VII.

Remarks.—This popular face salve, commonly called *cold-cream*, has the one drawback of rapidly turning rancid; hence the demand for a *theatrical cold-cream* for use by actors for cleansing the face from paints, and which will keep perfectly.

This is solved by the substitution of liquid petrolatum for the expressed oil of almond in the official recipe, though it is needless to say such a cold-cream should not be used in prescriptions.

UNGUENTUM BELLADONNÆ. Belladonna Ointment.

<i>Recipe.</i> —Extract of Belladonna Leaves, ten grammes....	10 Gm.
Diluted Alcohol, five cubic centimeters.....	5 Cc.
Hydrous Wool-Fat, twenty grammes.....	20 Gm.
Benzoinated Lard, sixty-five grammes.....	65 Gm.
To make about one hundred grammes..	100 Gm.

Triturate the Extract with the Diluted Alcohol until a smooth mixture is obtained; with this incorporate the Hydrous Wool-Fat; then add the Benzoinated Lard and mix thoroughly.

Remarks.—In preparing this ointment the extract is softened by trituration with diluted alcohol and then incorporated with hydrous wool-fat and benzoinated lard. The wool-fat is used to facilitate the absorption of the alkaloids of the extract by the skin.

UNGUENTUM CHRYSAROBINI. Chrysarobin Ointment.

<i>Recipe.</i> —Chrysarobin, six grammes.....	6 Gm.
Benzoinated Lard, ninety-four grammes.....	94 Gm.
To make about one hundred grammes..	100 Gm.

Triturate the Chrysarobin with the Benzoinated Lard, previously melted, and heat the mixture on a water-bath with occasional stirring for twenty minutes; then strain and stir until it congeals.

Remarks.—This is made by digesting chrysarobin in melted lard on a water-bath for twenty minutes and then stirring until congealed.

UNGUENTUM DIACHYLON. Diachylon Ointment.

<i>Recipe.</i> —Lead Plaster, fifty grammes.....	50 Gm.
Oil of Lavender Flowers, one gramme.....	1 Gm.
Olive Oil, forty-nine grammes.....	49 Gm.
To make one hundred grammes..	100 Gm.

Melt the Lead Plaster by applying a gentle heat, add the Olive Oil, and mix thoroughly; then allow the mixture to cool, add the Oil of Lavender Flowers, and stir the ointment until it congeals. It should be prepared extemporaneously.

Remarks.—This is lead plaster, thinned with olive oil and flavored with oil of lavender.

UNGUENTUM GALLÆ. Nutgall Ointment.

<i>Recipe.</i> —Nutmeg, in very fine powder, twenty grammes....	20 Gm.
Ointment, eighty grammes.....	80 Gm.
To make one hundred grammes..	100 Gm.

Rub the Nutgall with the Ointment, gradually added, until they are thoroughly mixed. Avoid the use of metallic utensils.

Remarks.—Like ointment of tannic acid, and for the same reason, metallic utensils must not be used in making this ointment.

UNGUENTUM HYDRARGYRI. Mercurial Ointment.

<i>Recipe.</i> —Mercury, five hundred grammes.....	500 Gm.
Oleate of Mercury, twenty grammes.....	20 Gm.
Prepared Suet, two hundred and thirty grammes...	230 Gm.
Benzoinated Lard, two hundred and fifty grammes.	250 Gm.
To make one thousand grammes..	1000 Gm.

Triturate the Oleate of Mercury in a warm mortar, add the Mercury gradually by means of a pipette, and when the globules are completely divided and distributed, set it aside for about fifteen minutes. Melt the Lard and Suet, allow the mixture to partially cool, and add about *twenty-five grammes* of it to the mercurial mixture, and continue the trituration until globules are no longer visible under a lens magnifying ten diameters. Then add the remainder of the Lard and Suet and mix thoroughly.

Remarks.—Mercurial ointment is prepared on the same principle as blue mass, in that the mercury is finely divided and prevented from running together by coating each minute globule. In this ointment the mercury is extinguished with oleate of mercury and the globules are covered with lard and suet. Note that the globules must not be visible under a microscope magnifying ten diameters.

This 50 per cent. ointment is no longer called blue ointment, a diluted 33 per cent. form having been introduced at the last revision for dispensing under that synonym. (See p. 347.)

UNGUENTUM HYDRARGYRI AMMONIATI. Ointment of Ammoniated Mercury.

<i>Recipe.</i> —Ammoniated Mercury, in very fine powder, ten grammes.....	10 Gm.
White Petrolatum, fifty grammes.....	50 Gm.
Hydrous Wool-Fat, forty grammes.....	40 Gm.
To make one hundred grammes..	100 Gm.

Rub the Ammoniated Mercury with an equal weight of the melted White Petrolatum, then add the remainder of the melted White Petrolatum, mix thoroughly with the Hydrous Wool-Fat, and stir the mixture until it congeals.

Remarks.—This preparation—called *white precipitate ointment*—made partly by fusion and partly by incorporation; that is, the ammoniated mercury is rubbed with melted petrolatum and then with unmelted hydrous wool-fat. Petrolatum alone is but slightly absorbed by the skin. Combined with wool-fat it is fairly absorbable.

UNGUENTUM HYDRARGYRI DILUTUM. Blue Ointment.

<i>Recipe.</i> —Mercurial Ointment, six hundred and seventy grammes.....		670 Gm.
Petrolatum, three hundred and thirty grammes.....		330 Gm.
To make one thousand grammes..		1000 Gm.

Mix them thoroughly.

Remarks.—This new official was introduced to give pharmacopœial sanction to the custom of dispensing a 33 per cent. mercurial ointment on calls for “blue ointment.” It is commercially called “mercurial ointment $\frac{1}{3}$.”

UNGUENTUM HYDRARGYRI NITRATIS. Ointment of Mercuric Nitrate.

Recipe and details of manipulation and full explanation of the chemistry of this important preparation will be found in Part VII.

UNGUENTUM HYDRARGYRI OXIDI FLAVI. Ointment of Yellow Mercuric Oxide.

<i>Recipe.</i> —Yellow Mercuric Oxide, in very fine powder, ten grammes.....		10 Gm.
Water, ten grammes.....		10 Gm.
Hydrous Wool-Fat, forty grammes.....		40 Gm.
Petrolatum, forty grammes.....		40 Gm.
To make one hundred grammes..		100 Gm.

Triturate the Yellow Mercuric Oxide with Water until the mixture is perfectly smooth, then add the Hydrous Wool-Fat in divided portions, and incorporate thoroughly with the Petrolatum, avoiding contact with metallic utensils.

Remarks.—Made by levigating the oxide with water and then triturating with wool-fat and petrolatum.

UNGUENTUM HYDRARGYRI OXIDI RUBRI. Ointment of Red Mercuric Oxide.

Recipe.—Identical with that for ointment of yellow mercuric oxide, except for use of red mercuric oxide (10 per cent., as in the case of the other chemical), and the directions to “triturate until absolutely free from gritty particles.”

UNGUENTUM IODI. Iodine Ointment.

<i>Recipe.</i> —Iodine, four grammes.....		4 Gm.
Potassium Iodide, four grammes.....		4 Gm.
Glycerin, twelve grammes.....		12 Gm.
Benzoinated Lard, eighty grammes.....		80 Gm.
To make one hundred grammes..		100 Gm.

Triturate the Iodine and Potassium Iodide in a glass mortar with the Glycerin until dissolved, then gradually incorporate the Benzoinated Lard and mix thoroughly, avoiding the use of a metallic spatula. This ointment should be freshly made when required.

Remarks.—The iodine in this preparation, as in the compound solution of iodine, is made soluble by the use of potassium iodide. Note that the rubber-coated spatula should be used for mixing.

UNGUENTUM IODOFORMI. Iodoform Ointment.

<i>Recipe.</i> —Iodoform, in very fine powder, ten grammes.....	10 Gm.
Lard, ninety grammes.....	90 Gm.
To make one hundred grammes..	100 Gm.

Triturate the Iodoform thoroughly with about twice its weight of the Lard, then incorporate the remainder of the Lard.

Remarks.—The odor of the iodoform can be removed from the mortar by burning alcohol therein (p. 270). Masking the odor is given under Iodoform.

UNGUENTUM PHENOLIS. Ointment of Phenol.

<i>Recipe.</i> —Phenol, three grammes.....	3 Gm.
White Petrolatum, ninety-seven grammes.....	97 Gm.
To make one hundred grammes..	100 Gm.

To the melted White Petrolatum add the Phenol, and stir the mixture until it begins to congeal.

Remarks.—The preparation commonly known as *carbolic salve* had its official name changed at the last revision, following the change of name of chemical from carbolic acid to phenol.

Some pharmacists still dispense as carbolic salve *ointment of carbolic acid* (U. S. P. 1890), which contained 5 per cent. phenol with Ointment (U. S. P. 1890).

UNGUENTUM PICIS LIQUIDÆ. Tar Ointment.

<i>Recipe.</i> —Tar, five hundred grammes.....	500 Gm.
Yellow Wax, one hundred and fifty grammes.....	150 Gm.
Lard, three hundred and fifty grammes.....	350 Gm.
To make one thousand grammes..	1000 Gm.

Melt the Yellow Wax, add the Lard, and to the melted mixture add the Tar, previously warmed, and incorporate thoroughly; strain through muslin, and stir the mixture until it congeals.

UNGUENTUM POTASSII IODIDI. Ointment of Potassium Iodide.

<i>Recipe.</i> —Potassium Iodide, ten grammes.....	10.0 Gm.
Potassium Carbonate, six-tenths of a gramme.....	0.6 Gm.
Water, ten grammes.....	10.0 Gm.
Benzoinated Lard, eighty grammes.....	80.0 Gm.
To make about one hundred grammes..	1000 Gm.

Dissolve the Potassium Iodide and Potassium Carbonate in the Water by trituration, then gradually add the Benzoinated Lard, and incorporate thoroughly. This Ointment should be prepared extemporaneously.

Remarks.—This ointment contains a small quantity of potassium carbonate, it being added to prevent the liberation of iodine from the iodide; the benzoinated lard, on becoming old, generating a sufficient quantity of acid to decompose the iodine, and if potassium carbonate is not present, the ointment will become brown.

UNGUENTUM STRAMONII. Stramonium Ointment.

Recipe.—Identical with that for belladonna ointment except the use of extract of stramonium.

Both ointments contain 10 per cent. of the characteristic extract.

Remarks.—This ointment is not the same as the ointment of stramonium (U. S. P. 1890), which was made from extract of stramonium seed.

UNGUENTUM SULPHURIS. Sulphur Ointment.

Recipe.—Washed Sulphur, one hundred and fifty grammes.. 150 Gm.

Benzoinated Lard, eight hundred and fifty grammes 850 Gm.

To make one thousand grammes.. 1000 Gm.

Rub the Washed Sulphur with the Benzoinated Lard, gradually added, until they are thoroughly mixed.

Remarks.—The ointment of the present pharmacopœia contains just one-half as much sulphur (15 per cent.) as did the ointment of the pharmacopœia of 1890.

UNGUENTUM VERATRINÆ. Veratrine Ointment.

Recipe.—Veratrine, four grammes..... 4 Gm.

Expressed Oil of Almond, six grammes..... 6 Gm.

Benzoinated Lard, ninety grammes..... 90 Gm.

To make one hundred grammes.. 100 Gm.

Rub the Veratrine with the Expressed Oil of Almond, then gradually add the Benzoinated Lard, and mix thoroughly.

Remarks.—This is interesting as the only official ointment made with an alkaloid. In its preparation the veratrine is made to a smooth paste with expressed oil of almond, benzoinated lard being then incorporated in the mixture.

UNGUENTUM ZINCI OXIDI. Ointment of Zinc Oxide.

Recipe.—Zinc Oxide, in very fine powder, two hundred grammes.....

200 Gm.

Benzoinated Lard, eight hundred grammes..... 800 Gm.

To make one thousand grammes.. 1000 Gm.

Rub the Zinc Oxide, which must be free from gritty particles, with an equal weight of the melted Benzoinated Lard, and with this incorporate the remainder of the Benzoinated Lard, previously melted; if necessary, strain the Ointment while warm, and stir thoroughly until it congeals.

Remarks.—In order to make this ointment (*zinc salve*) perfectly smooth and absolutely free from grit it is necessary, first of all, that the zinc oxide be the pure official product, and not the gritty white zinc oxide which sells at low prices as a pigment. (See p. 524.)

UNGUENTUM ZINCI STEARATIS. Ointment of Zinc Stearate.

<i>Recipe.</i> —Zinc Stearate, in fine powder, fifty grammes.....	50 Gm.
White Petrolatum, fifty grammes.....	50 Gm.
To make one hundred grammes..	100 Gm.

To the White Petrolatum, melted on a water-bath, add the Zinc Stearate. Continue the heat until the mixture becomes smooth, then stir while cooling, until it congeals.

Remarks.—This is a new official of considerable value in some forms of skin trouble.

Dispensing Ointments.—The finishing and dispensing of ointments and cerates is a matter requiring attention. In the first place, it should be borne in mind that the ointments, especially those made of benzoinated lard, have a tendency to become rancid, and such rancid ointments should never be dispensed; hence incorporated ointments should be prepared freshly as required. Nor should an ointment be dispensed containing the slightest quantity of grittiness, and to prevent this it is necessary to rub the medicinal substance thoroughly with a small quantity of the fatty matter before incorporating the



Fig. 206.—Tin ointment box.



Fig. 207.—Ointment jar.

entire amount. This applies particularly to the ointments containing either of the two oxides of mercury, both of which are usually applied in cases of granulated eyelids. If any of the particles of mercuric oxide be still in the gritty form, it is sure to produce irritation. Hence the necessity of an absolutely smooth ointment.

As to the dispensing of the ointment, it should never be sent out in a wooden box unless the wood of which it is made is prepared so as to be impervious to grease. Even in dispensing small quantities of ointment over the counter a wooden box should never be used, as the grease soon penetrates the wood, and the purchaser has a greasy package, which is an abomination. For small retail sales the pressed tin box (Fig. 206) should be used exclusively.

In dispensing ointments on prescriptions one of the several varieties of ointment jars (Fig. 207) should be used. These usually consist of a glass jar with a metallic cover fastening to jar by a screw-joint. The cover is nickel plated, and the label should be pasted to the side of the glass jar. If it is desirable to use a round label on the cover,

the form of ointment box shown in Fig. 208 should be used. When the label is placed on the side of the glass jar it invariably becomes greasy while in the cover just mentioned it is protected on top by a transparent sheet and underneath by card-board impervious to grease, and the label will, therefore, remain fresh for a long time. A recent and rather satisfactory way of dispensing ointments is in collapsible tubes (Fig. 209), being especially convenient for the person who expects to carry an ointment around with him. In this connection it might be well to cite a novel method of dispensing a mercurial ointment now in vogue, which consists in placing in soft gelatin capsules sufficient of the ointment for one rubbing, the set of capsules being carried around in a pill box in the vest-pocket. Old-time pharmacists, after placing the ointment in an ointment box, invariably "finished" them by ornamenting the surface exposed to the air with fanciful designs. These designs were made with specially cut pieces

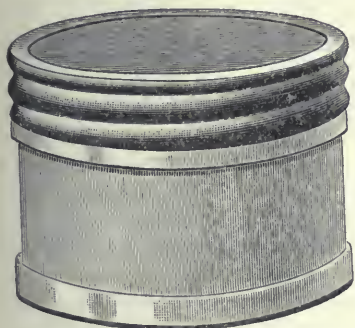


Fig. 208.—Ointment jar with mica top.



Fig. 209.—Collapsible tube.

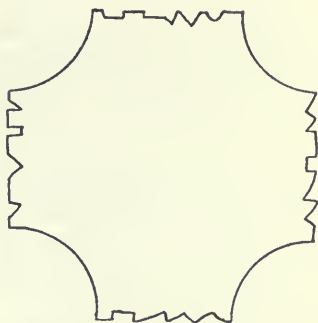


Fig. 210.—Tin ointment finisher.

of tin (Fig. 210), which was held firm while the ointment in the jar was gradually rotating under it. This idea was admirable in showing the purchaser whether the ointment had been tampered with by the messenger, but is now rapidly becoming obsolete, the pharmacist and purchaser depending more on the external appearance of the package for signs of tampering.

Preservation of Ointments.—Ointments should be stored in a cool place, preferably in a refrigerator.

CATAPLASMS

Cataplasms or *poultices* are wet masses of solid matter applied to the skin for the purpose of reducing inflammation, or in other cases to act as counterirritants.

The solid matter used as the base is chosen with a view to its capacity to absorb water. Thus the base of the one official cataplasm is clay, while mucilaginous drugs, such as ground flaxseed, are valuable as poultice bases.

If the poultice is intended to reduce inflammation, the proper base is one devoid of medicinal action, and the poultice wet with cold liquid acts similarly to a cold compress (that is, a wad of cloth or

cotton wet with water). If intended to act as a counterirritant, the poultice is either applied hot or is made from some drug possessing rubefacient properties. An illustration of the latter case is formed in the poultice made of ground mustard, which is replaced in the pharmacopœia by the more elegant *mustard paper*.

The one official cataplasm—that of *Kaolin*—belongs to the class of mechanical, non-medicinal poultices used for allaying inflammation.

CATAPLASMA KAOLINI. Cataplasm of Kaolin.

<i>Recipe</i> .—Kaolin, in very fine powder, five hundred and seventy-seven grammes.....	577.0 Gm.
Boric Acid, in very fine powder, forty-five grammes.....	45.0 Gm.
Thymol, one-half gramme.....	0.5 Gm.
Methyl Salicylate, two grammes.....	2.0 Gm.
Oil of Peppermint, one-half gramme.....	0.5 Gm.
Glycerin, three hundred and seventy-five grammes...	375.0 Gm.
To make about one thousand grammes..	1000.0 Gm.

Heat the Kaolin in a suitable vessel at 100° C. (212° F.), with occasional stirring, for one hour; mix it intimately with the Boric Acid, and then incorporate the mixture thoroughly with the Glycerin; finally add the Thymol, which has been dissolved in the Methyl Salicylate and the Oil of Peppermint, and make a homogeneous mass. It should be kept in an air-tight container.

Remarks.—This new official, which consists of a mixture of kaolin, boric acid, and glycerin, flavored with thymol, methyl salicylate, and oil of peppermint, is similar to a widely popular proprietary preparation largely prescribed by physicians. It is indicated as a local application in a large number of inflammatory diseases, and is also of value as a mild antiseptic in minor wounds.

PLASTERS

Plasters are solid preparations containing medicinal substances intended to be applied to the skin, and of sufficient adhesiveness to adhere firmly. Plasters are different from cerates in being practically free from fats, and also in the fact that the cerates, when spread on cloth and applied, are not of sufficient adhesiveness to stick firmly to the skin, their removal being a very simple matter. Plasters, on the other hand, made of adhesive or resinous substances, stick very firmly to the skin; in fact, so firmly that it is sometimes a matter of difficulty to remove them.

As bases of plasters are used gum-resins, lead-plaster, resin plaster, Burgundy pitch, isinglass, and, latterly, India rubber. In former years the first-named plaster bases were used quite extensively, but during the last twenty years machine-made plasters with rubber base have practically driven the old-fashioned type of plasters out of the market, so that now the pharmacist is very rarely called upon to dispense a pharmacopœial plaster, and the art of spreading plasters is rapidly becoming a lost one.

Before discussing the official plaster masses a few words regarding the manufacture of rubber plasters may not come amiss.

The original and really satisfactory India-rubber plasters are made by kneading crude rubber with resinous substances, such as gum

olibanum, until a plastic mass results. This kneading is accomplished by rolling the rubber between spiked hollow rollers heated by steam, the combination of the heat and the tearing effect of the spikes having the result of disintegrating the rubber into a thick, tenacious mass, with which the olibanum and other resinous mass combine admirably. The plaster mass is then spread on cloth by rolling between hot plates, both heat and pressure being required to spread it to the proper thickness. The making of such rubber plasters requires powerful and expensive machinery, and scarcely had the rubber plaster manufacture been placed on a paying basis, before other and inferior methods were substituted for the original process. In the cheaper methods the rubber is dissolved in the appropriate solvent, usually benzin, and the solution evaporated down to the consistence of a soft extract; and when it is spread on cloth, the surplus benzin is permitted to evaporate.

Such plasters are in every way inferior to the machine-kneaded plasters; the cohesive property of the mass is less, and such a plaster, on removal from the back to which it has been applied, very frequently tears off, leaving patches of the plaster sticking to the skin.

Of the pharmacopœial bases, a description had best be left for consideration under Special Notes.

The manufacture of pharmacopœial plasters may be divided into two parts: making the mass and spreading the same. The making of the official plaster mass is usually a matter of little difficulty, being generally prepared by fusing the various constituents or, in some cases, by softening them by gentle heat. The official plaster mass is then rolled into appropriate sticks, or left in the plaster dippers in which they have been fused, until a spreaded plaster is called for, the pharmacopœia directing in all cases, save one, merely the manufacture of the plaster mass, the spreading being left to the order of the physician and to the skill of the pharmacist. The one plaster specifically directed by the pharmacopœia to be spread is capsicum plaster.

The spreading of a plaster is a matter calling for a high degree of skill and requiring much practice. Full details of the spreading of a plaster will be given in Part VII. So here suffice it to say that the molten plaster mass is spread on plaster skin or cloth, the area, including the dimensions directed by the physician, being inclosed in strips of paper, the entire paraphernalia—the cloth or skin base and the paper—being firmly fastened to a perfectly level table or to a smooth board resting on the counter by means of thumb tacks. So soon as the molten plaster mass is poured upon cloth or skin base, it is spread by the rapid movement of a hot, clean spatula. If, in spreading, the hand trembles or the plaster mass is not warm enough to follow the spatula, an unsuccessful plaster results.

In former years there was considerable demand for specially shaped plasters, such as those to fit behind the ear, to cover the breast, or to place over the kidneys. These were spread on cloth by substituting for the plain paper strips above mentioned a pattern made of stiff paper, having cut out from its center the shape required for the plaster (Figs. 211, 212, and 213). The details of the spreading of the specially shaped plasters are exactly the same as for an oblong plaster.

In the old times, when plaster spreading flourished and numbers of plasters were called for in a day's prescription business, special



Fig. 211.—Ear plasters: *a*, For left ear; *b*, for right ear (Thornton).



Fig. 212.—Breast plaster (Thornton).



Fig. 213.—Kidney plaster (Thornton).

apparatus was devised for spreading plasters by hand on a large scale. An example of these spreading machines is found in the plaster spreader, a picture of which is appended (Fig. 214).



Fig. 214.—Plaster spreader.

At the present time there are few stores possessing such, the demand for hand-spread plasters being practically nil. Among the few survivors of hand-spread plasters are the so-called fly-blisters (p. 341).

The obsolescence of plasters is shown in the large number of plaster masses dropped in the last pharmacopœial revision. While thirteen plaster masses were recognized in the pharmacopœia of 1890, but seven are official in the present pharmacopœia. Two radical changes affecting plasters

were made at the last revision; the change in the recipe of lead plaster and the introduction of a rubber adhesive plaster base.

TABLE OF OFFICIAL PLASTERS (ALL GALENIC EXCEPT LEAD PLASTER)

<i>Process.</i>	<i>Base.</i>	<i>Preparations.</i>
By chemical decomposition (precipitation).	Soap and lead acetate.	Lead plaster.
By fusion.	Lead plaster.	Adhesive plaster. Mercurial plaster. Soap plaster. Belladonna plaster. Opium plaster.
Spread plaster.	Adhesive plaster.	Capsicum plaster.

SPECIAL NOTES ON OFFICIAL PLASTERS

EMPLASTRUM ADHÆSIVUM. Adhesive Plaster.

<i>Recipe.</i> —Rubber, cut in small pieces, twenty grammes.	20 Gm.
Petrolatum, twenty grammes.	20 Gm.
Lead Plaster, nine hundred and sixty grammes.	960 Gm.
To make one thousand grammes.	1000 Gm.

Melt the Rubber at a temperature not exceeding 150° C. (302° F.); add the Petrolatum, and continue the heat until the Rubber is dissolved. Add the Lead Plaster to the hot mixture; continue the heat until it becomes liquid, then strain, allow it to cool, and stir until it stiffens.

Remarks.—This new official is the first attempt of the United States Pharmacopœia to introduce a rubber plaster base. The amount of rubber (2 per cent.) is quite small, however, and therefore quite different from the rubber-base medicinal plasters of the manufacturer. It is, however, similar to the commercial rubber adhesive plaster (*sticking plaster*) which is spread by machinery on a large scale.

The apparatus consists of reels on one of which the original bolt of cloth is placed; the unrolled cloth passing between the lower plate of stone or metal, and a receptacle containing the melted plaster mass; one side of the receptacle consists of a dull steel blade resting $\frac{1}{8}$ or $\frac{1}{4}$ inch above the slightly stretched cloth. The melted mass trickles upon the cloth in a broad stream, covering the entire width of the cloth, save a small marginal edge. As the soft mass flows upon the cloth it is rapidly passed between the spreading apparatus described above, and emerges from the same with a thin and uniform coating of the plaster material.

EMPLASTRUM BELLADONNÆ. Belladonna Plaster.

Belladonna Plaster should contain not less than 0.38 per cent. nor more than 0.42 per cent. of mydriatic alkaloids.

Recipe.—Extract of Belladonna Leaves, three hundred grammes.....

300 Gm.

Adhesive Plaster, seven hundred grammes.....

700 Gm.

To make about one thousand grammes.. 1000 Gm.

Melt the Adhesive Plaster on a water-bath, add to it the Extract of Belladonna Leaves, softened by the heat of a water-bath, and continue the heat, stirring constantly until the mixture is perfectly homogeneous; then allow it to cool.

Spread Belladonna Plasters made with a rubber base should yield, when assayed, not less than 0.38 per cent. nor more than 0.42 per cent. of mydriatic alkaloids.

Remarks.—This plaster mass is made by combining extract of belladonna with adhesive plaster. This is the only plaster directed by the pharmacopœia to be of assayed strength— $\frac{3.8}{100}$ to $\frac{4.2}{100}$ of 1 per cent. mydriatic alkaloids. Details of assay will be given in Chapter LV.

EMPLASTRUM CAPSICI. Capsicum Plaster.

Recipe.—Oleoresin of Capsicum, twenty-five hundredths of a gramme.....

0.25 Gm.

Adhesive Plaster, spread on fabric, a sufficient quantity.

Apply the Oleoresin of Capsicum to the surface of the Adhesive Plaster by means of a brush, so as to form a thin coating over an area fifteen centimeters square, leaving a margin around the sides.

Remarks.—As already mentioned, this is the one plaster in the official recipe of which are given explicit directions as to spreading, the object being uniformly to distribute the oleoresin of capsicum as well as to limit the amount of this irritating substance employed. The oleoresin is chosen in preference to the drug because the latter, blended with the plaster base, would soon lose its activity. For this reason these plasters should be dispensed freshly prepared.

EMPLASTRUM HYDRARGYRI. Mercurial Plaster.

<i>Recipe.</i> —Mercury, thirty grammes.....	30 Gm.
Oleate of Mercury, one gramme.....	1 Gm.
Hydrous Wool-Fat, ten grammes.....	10 Gm.
Lead Plaster, fifty-nine grammes.....	59 Gm.
To make one hundred grammes..	100 Gm.

Triturate the Mercury with the Oleate of Mercury until the former is thoroughly divided, then add the Hydrous Wool-Fat, and continue the trituration until globules of Mercury are no longer visible. Add the mixture to the Lead Plaster, which has previously been melted in a tared dish, and incorporate thoroughly, adding, if necessary, sufficient Lead Plaster to make the product weigh *one hundred grammes*.

Remarks.—In this plaster the mercury is extinguished with oleate of mercury and hydrous wool-fat and then mixed with lead plaster.

EMPLASTRUM OPII. Opium Plaster.

<i>Recipe.</i> —Extract of Opium, six grammes.....	6 Gm.
Water, eight cubic centimeters.....	8 Cc.
Adhesive Plaster, ninety grammes.....	90 Gm.
To make one hundred grammes..	100 Gm.

Rub the Extract of Opium with the Water until it is uniformly soft; add it to the Adhesive Plaster, which has been previously melted in a tared dish on a water-bath, and continue the heat with constant stirring until the product weighs *one hundred grammes*.

Remarks.—This plaster contains 6 per cent. of opium mixed with adhesive plaster.

EMPLASTRUM PLUMBI. Lead Plaster.

Recipe and details of manufacture of this important plaster base, as well as full explanation of the chemistry of manufacture, will be given in Part VII.

EMPLASTRUM SAPONIS. Soap Plaster.

<i>Recipe.</i> —Soap, dried, and in a coarse powder, ten grammes..	10 Gm.
Lead Plaster, ninety grammes.....	90 Gm.
Water, a sufficient quantity,	
To make about one hundred grammes..	100 Gm.

Rub the Soap with enough Water to reduce it to a semiliquid state; then mix it with the Lead Plaster previously melted, incorporate thoroughly by stirring, and evaporate to the proper consistence.

Remarks.—This plaster mass is lead plaster softened by addition of 10 per cent. soap.

Of the plasters dropped at the last revision, but two are worthy of notice.

EMPLASTRUM ICHTHYOCOLLÆ (U. S. P. 1890). Isinglass Plaster.

This is made by spreading aqueous solution of isinglass by means of a brush upon taffeta, successive layers of isinglass being applied to this fabric (a variety of silk) until the coating is sufficiently thick.

The last portion of isinglass solution is mixed with alcohol and glycerin and applied in the same manner. The back of the taffeta is then spread with tincture of benzoin, and the official plaster allowed to become perfectly dried. Isinglass plaster is the well-known *court-plaster*, and is intended for adhesive purposes in simple operations, such as bringing the two edges of open cuts together. It has no medical action.

EMPLASTRUM PICIS BURGUNDICÆ. Burgundy Pitch Plaster.

This is made by softening Burgundy pitch with yellow wax and olive oil, the Burgundy pitch being so brittle that, if melted and spread on cloth by itself and allowed to become quite cold, the finished plaster will break very quickly. Burgundy pitch plasters formerly enjoyed a large sale under the name of "*poor man's plasters*."

PAPERS

Papers are a class of pharmaceuticals in which the medicine is either spread upon paper or absorbed by it.

But one paper is recognized by the present pharmacopœia.

CHARTA SINAPIS. Mustard Paper.

Recipe.—Black Mustard, in No. 60 powder, *one hundred grammes*..... 100 Gm.
 Rubber, *ten grammes*..... 10 Gm.
 Petroleum Benzin,
 Carbon Disulphide, each, *a sufficient quantity*.

Pack the Black Mustard in a conical percolator, and gradually pour Petroleum Benzin upon it until the percolate ceases to produce a permanent, greasy stain upon blotting paper. Remove the powder from the percolator, and dry it by exposure to the air. Having meanwhile dissolved the Rubber in a mixture of *one hundred cubic centimeters*, each, of Petroleum Benzin and Carbon Disulphide, mix the purified mustard with a sufficient quantity of the solution to produce a semiliquid magma, and apply this, by means of a suitable brush, to one side of a piece of rather thick, well-sized paper, so as to cover it completely, and then allow the surface to dry.

A surface of sixty square centimeters should contain about 4 Gm. of Black Mustard deprived of oil.

Before it is applied to the skin, Mustard Paper should be dipped in warm water for about fifteen seconds.

Remarks.—This paper is prepared by mixing powdered black mustard (which has been freed from oil by percolation with benzin) with a solution of India rubber in benzin and carbon disulphide, and spreading the semiliquid magma on a piece of stiff well-sized paper (by means of a suitable brush), the pharmacopœia requiring that 60 square centimeters of such paper should contain about 4 Gm. of black mustard deprived of oil. The object of first extracting the mustard with benzin is to free it from the fixed oil naturally existing in the seed, and which, if permitted to remain in the mustard paper, would rapidly cause it to become greasy and disagreeable to handle. Mustard paper (*mustard leaves*, as they are called) affords an elegant and convenient method of applying the mustard plaster. When such a mustard leaf is dipped into warm water, a chemical action sets up between the ferment myrosin and the glucoside potassium myronate,

naturally existing in the mixture, with the formation of the volatile oil of mustard (see p. 747). The pharmacopœia directs that the leaf be dipped in warm water, since dipping it in cold water retards the chemical action to a certain extent. On the other hand, it is far preferable to dip into cold water than into hot water, and under no circumstances should the mustard paper be moistened with boiling water, as the heat is sufficient to destroy the delicate ferment, myrosin, and thus prevent the formation of the volatile oil.

Mustard leaves are applied externally as a rubefacient and local irritant, being particularly adapted for cases of acute indigestion.

There was one other paper recognized by the pharmacopœia of 1890.

CHARTA POTASSII NITRATIS (U. S. P. 1890). Potassium Nitrate Paper.

This is made by dipping white, unsized paper (either filter-paper or blotting-paper) into an aqueous solution of potassium nitrate, and drying the pieces so dipped. The potassium nitrate paper is intended to be burnt and the vapor inhaled in cases of asthma.

PART III

CHAPTER XXII

INTRODUCTION

A THOROUGH explanation of the sciences of physics and chemistry is beyond the scope of this work, and for complete information on these two sciences the reader is referred to the several works on these subjects noted in the Preface to this work.

There are, however, some facts connected with these two sciences the knowledge of which is essential to a student of the succeeding chapters of this work, and these essential facts are here given.

Matter is anything that has extension (possesses length, breadth, and thickness) or is appreciable to our senses (can be seen, felt, tasted, or smelled). Thus, water is matter, or anything else on this earth or in the air around us, which we can touch, which our senses tell us exist. All matter is divided into solids, liquids, and gases.

A *solid* is that form of matter which holds a definite shape—whose molecules do not move among themselves.

A *liquid* is that form which is mobile—which assumes the shape of the vessel in which it is placed; whose molecules move freely among themselves.

A *gas* is that form of matter whose molecules tend to separate from one another.

When we call any portion of matter a solid, a liquid, or a gas, we mean that it usually assumes the form which we call it. We know that water, usually a liquid, can be frozen into a solid—ice—or converted by heat into gas—steam. Such is the case with all other matter. Each kind of matter can be changed from the form it usually assumes—be it solid, liquid, or gas—into the other two forms, although sometimes the conversion is very difficult.

Thus, we formerly talked of “permanent gases,” by which was meant such gases—air, for instance—that could not be converted into the forms of liquid or solid; but lately not only have the so-called “permanent gases” been turned into liquids, but into solids as well.

The word “*molecule*,” used above, is one of the classes into which we group matter, according as we can divide it or break it up. A piece of coal can be taken and broken up into small pieces. These small pieces can be finely powdered, and by certain means we can divide it still more minutely. According to how far this division is carried on we group matter into masses, molecules, and atoms.

A *mass* is any particle of matter appreciable to our senses. Thus, a big lump of coal or the finest particle of its powder are alike masses.

A *molecule* is the smallest particle of matter than can exist in a free state.

Thus, if we take a dye, dissolve it in water, and keep on diluting the solution, the color grows fainter and fainter until finally we reach

a point where the tint almost disappears. It is at this point where we get the dye divided into its molecules. By this means Hoffmann found that one-seven-millionth of a gramme of dye in a cubic centimeter solution still had a pink tint.

We have never seen a molecule; we have never weighed a molecule—but in this way we can approximate how small a molecule is. Some say that a molecule of hydrogen weighs about one-five-billionth of a grain.

Finally, we can imagine a subdivision so minute that the dye is separated from the carbon, hydrogen, oxygen, and nitrogen of which it is composed. Then we have reached the atom.

An *atom* is the smallest particle which enters into a chemical combination. *An atom cannot exist in a free state.*

Now that we know what atoms and molecules are, we can talk about physics and chemistry. Among the many definitions of the two sciences, the simplest is this:

Physics treats of the *molecular* changes of bodies, while *chemistry* studies the *atomic* changes.

Thus, we heat an iron rod. It turns red and finally softens and glows with white light. But when we allow it to cool, we find it unchanged. In other words, the molecules were temporarily changed. The iron had undergone a *physical change*.

We take powdered iron and powdered sulphur and rub them together, when we get a grayish-yellow powder from which we can separate the iron with magnet or can see its minute particles through a microscope. Intimate though the incorporation may seem, we have merely a mechanical mixture, the molecules of the iron and of the sulphur being absolutely unchanged. We heat this mixture; not merely do the sulphur and the iron molecules undergo the physical change called heating, but the molecules are broken up, the atoms of which they are composed enter into the play, and, through the breaking of the old association and the formation of the new, we get a steel-gray solid, totally different from the iron and the sulphur of which it is composed, from which the iron can no longer be obtained by the magnet; in which we can no longer see the particles of iron by means of the microscope. We have a new compound—iron sulphide; for the molecules of iron and sulphur have broken up, their constituent atoms uniting in another combination. Such is a *chemical change*.

Many phenomena which we study in pharmacy are due to attractions exerted between atoms or molecules. Such attractions were formerly called forces, and we still speak of the "force of gravity," the "force of chemism," etc. The word "force" being now, however, used in a more restricted sense in physics, its application in the above cases is no longer usual.

Important among these attractions are cohesion, adhesion, and chemism.

Cohesion is that attraction exerted between *like* molecules. Thus, when we take two pieces of freshly cut lead and press them together they stick. Likewise a huge mass of lead holds together and does not fall to powder because its molecules cohere.

Adhesion is the attraction exerted between *unlike* molecules. Thus, in sticking a label on a bottle the molecules of the mucilage

adhere to the molecules of the paper on one side and of the glass on the other.

Chemism is the attraction exerted between atoms. It acts only *within* the molecule, and is exerted only between definite weights of matter.

We have spoken of carbon, hydrogen, oxygen, nitrogen, sulphur, and iron. What are such kinds of matter? You will notice that we have spoken of *atoms* of these kinds of matter; so you can see that they are capable of very fine division.

Let us think of a mass of sea-water. If we attempt to separate it into its constituents, we will find water, salt, magnesium chloride, and many other bodies. Suppose we analyze the salt it contains. This we can divide into sodium and chlorine. Turning our attention to the separated sodium and chlorine: can we break them up as we have the salt or the sea-water? We cannot, and such matter that cannot be broken into anything simpler we call *elements*.

An element is matter that cannot be broken into anything simpler. Over seventy elements have been isolated, and of these, less than half are of interest in pharmacy.

A table of those of pharmaceutic importance will be found on p. 367, each accompanied by its symbol, atomic weight, and valence, and grouped in one of the two columns according as it is electro-positive or electronegative.

By *symbol* we mean an abbreviation representing the element.

The abbreviation is usually the first letter of the Latin name of the element, but when that letter is already the symbol of an element, then a second letter is added to make the symbol distinctive.

Thus:

	LATIN.	SYMBOL.
Carbon.....	Carbo.....	C.
Calcium.....	Calcium.....	Ca.
Cerium.....	Cerium.....	Ce.
Cobalt.....	Cobaltum.....	Co.
Copper.....	Cuprum.....	Cu.
Columbium.....	Columbium.....	Cb.
Cadmium.....	Cadmium.....	Cd.
Chlorine.....	Chlorum.....	Cl.
Chromium.....	Chromium.....	Cr.
Cæsium.....	Cæsium.....	Cs.

This symbol does not merely mean the element in question in a general way, but when written, indicates the quantity of the element. Thus Ca means one atom of calcium. If we desire to express any other number of atoms, we do so by placing below and to the right of the symbol a small Arabic number representing that quantity.

Thus, while Ca means one atom of calcium

Ca₂ means two atoms of calcium

Ca₃ means three atoms of calcium, etc.

By *symbolic formula* we mean a combination of symbols expressing the composition of a compound molecule. Thus, NaCl means one atom of sodium and one atom of chlorine, making together one molecule of sodium chloride.

H_2SO_4 means two atoms of hydrogen, one atom of sulphur, and four atoms of oxygen, making together one molecule of sulphuric acid.

KNO_3 means one atom of potassium, one atom of nitrogen, and three atoms of oxygen, making together one molecule of potassium nitrate.

ATOMIC WEIGHT

The atomic weight of an element is the *relative combining weight of that element* when compared to hydrogen—the lightest of all substances—as a unit.

Elements combine with each other only in definite proportions. Thus, it has been proved, time and again, that if 63 Gm. of pure copper be heated in a stream of oxygen, 79 Gm. of copper oxide invariably result. This shows that the relative combining weights of copper and oxygen are respectively 63 and 16 ($63 + 16 = 79$).

But how do the combining weights of these elements compare with hydrogen? Copper and hydrogen scarcely unite. Oxygen and hydrogen do; although the estimation of the combining proportions of the two is more complicated than that between copper and oxygen, and an explanation is needed.

It is found that *two* volumes of hydrogen gas (let us say, for example, two pints) unite with *one* volume of oxygen gas (say, one pint) to form water, and this is still better proved by the fact that water, decomposed by electricity, gives always *two* volumes of hydrogen to *one* of oxygen.

An Italian named Avogadro framed a law that equal volumes of different gases contained the same number of molecules. Then, if water is composed of two volumes of hydrogen and one volume of oxygen, and if, further, both hydrogen and oxygen contain two atoms in each molecule, as will be shown later, then the formula of water is H_2O and not HO .

Now, water is composed of two *volumes* of hydrogen and one *volume* of oxygen. By weight, however, it is made from the proportion of *one* gramme of hydrogen and *eight* grammes of oxygen. By uniting the two statements just made, you will see that, while there is in water only *half* as many volumes of oxygen as of hydrogen, the weight of that quantity of oxygen is eight times greater than the double volume of hydrogen employed. If half weighs eight times as much, then a whole (equal volume) would weigh sixteen, and we, therefore, see that were hydrogen and oxygen to unite volume for volume the relative combining weights would be 1 to 16. Since hydrogen is the lightest of all known matter, it is given the atomic weight, 1; therefore, we say that the atomic weight of oxygen is 16, and, by the same reasoning, that of copper is 63. By similar experimentation, some of which was exceedingly ingenious, the atomic weights of all the elements have been established.

Atomic weight is sometimes supposed to represent relative weight of an atom of the element under consideration, but, as we have never obtained a free atom of any element, that definition of atomic weight had better be dropped.

It will be noticed, in the table of elements given on p. 367, that the atomic weights are expressed in round numbers, while the atomic

weights given in the pharmacopœia and other standard works are figures with decimal fractions attached.

Thus, while the table on p. 367 gives the atomic weight of sulphur as 32, the pharmacopœia gives it as 31.98; while the table gives the atomic weight of oxygen as 16, the pharmacopœia gives it as 15.96, and so in many other cases. Space forbids a discussion of the controversy as to whether the "exact" atomic weights (the figures showing fractions or the weights expressed in round numbers) are the true atomic weights. Suffice it to say that in the table in this book the round numbers are given, first, because the memorization of the fractional numbers is a matter of difficulty,—and the table under consideration should be memorized,—and, second, the so-called "exact" atomic weights change year by year with the publication of new researches with the combining weights of the elements; moreover, for all the practical purposes of pharmacy, the round number atomic weights are sufficiently exact.

We have explained atomic weight, and it remains to define molecular weight.

The molecular weight of a substance is the sum of the atomic weights of its constituent atoms. Thus, the molecular weight of sodium chloride (NaCl) is:

Na, atomic weight.....	23.0	
Cl, “ 	35.5	
NaCl, molecular weight....	<u>58.5</u>	
 Of Ca Cl ₂ :		
Ca, atomic weight..	40	Ca = 1 × 40 = 40
Cl, “ 	<u>35.5</u>	Cl ₂ = 2 × 35.5 = 71
		Ca Cl ₂ , molecular weight, <u>111</u>

We have means of estimating molecular weight that are quite different from the process of finding the atomic weight of elements, and, as the molecular weight of elements found by such processes are almost always twice the atomic weight, we are led to believe that most elemental molecules contain two atoms.

Thus, the molecular weight of oxygen is 32; the atomic weight is 16; therefore a molecule of oxygen must be O_2 . On the other hand, while the atomic weight of mercury is 200, its molecular weight estimation gives the same figure, and we are, therefore, forced to the conclusion that a molecule of mercury consists of one atom.

Again, atomic weight estimation of phosphorus shows 31 as its relative combining weight, while its molecular weight has been experimentally proved to be 124; hence one molecule of phosphorus must contain four atoms.

VALENCE

Valence is the relative power which an element exerts in uniting with or replacing other elements. Thus, analysis of the four typical compounds cited below gives the formula there stated:

Hydrochloric acid.....HCl.
Water.....H₂O.
Ammonia gas.....H₃N (or NH₃).
Methane (marsh-gas)..H₄C (or CH₄).

Why is it that while chlorine unites with one atom of hydrogen, oxygen can take up two, nitrogen (N) three, and carbon (C) four atoms of hydrogen? Simply because carbon has four times the combining *power* as hydrogen or chlorine, and twice the combining *power* of oxygen. Valence has nothing to do with atomic weight. While the latter concerns itself over combining *weight*, valency refers only to combining *power*. That carbon has the valence four—has four times the combining power of hydrogen—is analogous to the fact that a four-dollar check would have four times the purchasing power of a one-dollar check. Akin to this is the illustration that a man with four hands could do four times as much grasping as a man with one hand. This brings up the graphic way of illustrating valence called after its originator, "Frankland's bonds."

Frankland represented valence after this idea of hands, sketching the symbol of the element with as many lines as its valency.

Thus:

Hydrogen with valence	one	is sketched as	H—
Chlorine	"	one	" Cl—
Oxygen	"	two	" O==
Nitrogen	"	three	" N===
Carbon	"	four	" C====

While the compounds cited above are given the following graphic formula:

Hydrochloric Acid.



Water.



Ammonia.



Methane.



To get the idea of valence imagine hydrogen and chlorine as one-armed men clasping hands; oxygen, with two hands going down the street with two single-handed hydrogen atoms; nitrogen as a three-handed being, and carbon as possessing four hands.

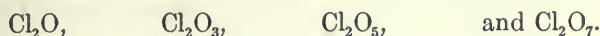
It is hardly necessary to state that this picture is purely fanciful; that this combining power must, in truth, consist of an atomic attraction somewhat similar to the attraction of gravitation as exerted between masses. Or perhaps that variation of combining power may be the proportional variation of electric manifestation.

In fact, there is some question among chemists in these days as to the entire theory of valence, and especially as to the varying valences exhibited by the same element, noted in the following pages.

But be this as it may, the fact still remains that the theory of valence affords the only rational and practical way of learning to write the symbolic formulæ of chemicals, and in this spirit the subject is discussed in these pages.

In the four compounds given as examples of valence, hydrogen plays a leading part, but when we consider the entire vast mass of chemical compounds we find that the hydrogen compounds are few, and that oxygen is the element preëminent for combinations. This is well shown in the combinations of chlorine. Hydrogen and chlorine unite to only one compound, the hydrochloric acid (HCl) given above. But when we look up the combinations of chlorine

and oxygen, either free or in combination, we find four different bodies having the following formulas:



We know that the valence of oxygen is two, that each atom has two bonds: then let us see how the linking power of chlorine varies, by analyzing the four formulas given above.

It is evident that the first, Cl_2O , which is analogous to H_2O , has as graphic formula $\text{Cl}-\text{O}-\text{Cl}$, and that the valence of chlorine is one.

Now for Cl_2O_3 . Three atoms of oxygen means six bonds to be divided between two atoms of chlorine. Therefore, each chlorine must take up three bonds of oxygen, giving the graphic formula



In Cl_2O_3 the valence of chlorine must be three.

In Cl_2O_5 the five oxygen atoms give us ten bonds, so each chlorine must make up five bonds, thus:



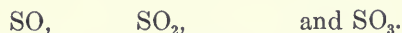
and the valence of chlorine is five.

In Cl_2O_7 the seven oxygen atoms mean fourteen bonds, with seven bonds for each chlorine to take up, thus:



and in this case the valence of chlorine must be seven.

Then, we have three combinations of sulphur, with oxygen:



In $\text{S}=\text{O}$ the valence of sulphur must be two.

In $\text{S}=\text{O}$ " " " four.

In $\text{S}=\text{O}$ " " " six.

This suggests that the valence of an element varies, and the variation has been explained in this manner: The true valence of an element is its highest. Thus, in sulphur the true valence is six,



In some way two of the "hands" (bonds) become clasped, and just as it is impossible to pick up anything with clasped hands, so those two bonds became useless for linkage purposes and the sulphur atom possesses but four active bonds, as shown below.



Again two more "hands" grasp—two bonds saturate each other, as the term is—and there remain but two active bonds to the sulphur atom, its valence is two, thus,



This fanciful picture is given for what it is worth, and, contrary to this theory, the usual and most stable valence is the lowest.

The valence of an element is expressed in Roman numerals above and to the right of its symbol; thus, chlorine is expressed as Cl^{I} , Cl^{III} , Cl^{V} , or Cl^{VII} respectively, according as its valence is one, three, five, or seven.

In the table of elements found on the following page not only is the valence and atomic weight of each element given, but the elements are grouped in two columns, according as they are electropositive or electronegative.

These two terms indicate the behavior of the elements when compounds containing them are subjected to the action of a current of electricity.

The subject of electricity is beyond the scope of this work, and here can mention be made only of the fact that electricity is developed by the dynamo, such as can be seen at any electric lighting station, and the battery, such as accompanies every telephone or telegraph instrument.

From every battery two wires emerge, and through these wires pass electricity of different quality, called positive and negative, and the termination of the two wires are called the positive pole and the negative pole respectively.

If we take common salt, sodium chloride, melt it and subject it, while molten, to a current of electricity, at the positive pole of the battery we get the odor of chlorine; while at the negative pole we get metallic sodium.

Now, the electric rule is: "Like electricities repel each other; unlike attract each other," and, since sodium goes to the negative pole, we call it positive; while chlorine, going to the positive pole, is called negative. Accordingly, we see that sodium chloride contains one portion that is positive and another that is negative, and, from this, Berzelius worked out his electromagnetic theory, by which he divided every chemical compound into positive and negative parts, and attempted to explain chemical action by electric influence. But his theory did not stand the test of time, and to-day but little remains but the fact that we speak of positive elements, to which we give the sign "+," and of negative elements, which are marked "-." All

the metals are found to be positive, while the non-metallic elements are negative. The division, however, is purely relative. Thus, when zinc is combined with chlorine it is the positive part of the compound; whereas, when combined with sodium it is the negative element.

ELEMENTAL TABLE

NEGATIVE.				POSITIVE.			
	Name.	Symbol and Valence.	Atomic Weight.	Name.	Symbol and Valence.	Atomic Weight.	
MONADS. (Usually having valence I.)	Chlorine....	Cl. I III V VII	35.5	Lithium.....	Li. I	7	
	Bromine....	Br. I III V VII	80.	Sodium.....	Na. I	23	
	Iodine.....	I. I III V VII	126.	Potassium....	K. I	39	
				Copper.....	Cu. I ("ous" form)	63	
				Silver.....	Ag. I	108	
				Mercury.....	Hg. I ("ous" form)	200	
DYADS. (Usually having two bonds— the valence II.)	Oxygen.....	O. II	16.	Magnesium...	Mg. II	24	
	Sulphur.....	S. II IV VI	32.	Calcium.....	Ca. II	40	
				Strontium....	Sr. II	87	
				Barium.....	Ba. II	137	
				Iron.....	Fe. II ("ous" form)	56	
				Copper.....	Cu. II ("ic" form)	63	
				Zinc.....	Zn. II	65	
				Mercury.....	Hg. II ("ic" form)	200	
				Lead.....	Pb. II	206	
				Tin.....	Sn. II ("ous" form)	118	
	TRIADS. (Having the val- ence III, usu- ally.)	Boron.....	B. III	11.	Aluminum...	Al. III	27
		Nitrogen.....	N. III V	14.	Iron.....	Fe. III ("ic" form)	56
Phosphorus...		P. III V	31.	Gold.....	Au. III	196	
Arsenic.....		As. III V	75.	Bismuth.....	Bi. III	207	
Antimony....		Sb. III V	120.	Cerium.....	Ce. III	140	
TETRADES. (Valence of IV.)	Carbon.....	C. IV	12.	Tin.....	Sn. IV ("ic" form)	118	
	Silicon.....	Si. IV	28.				

Chromium (Cr, at. wt. 52) and manganese (Mn, at. wt. 54.8), while found in official compounds, are omitted from the table for reasons given on pp. 528 and 541.

Following the theory of variation in valence cited on p. 365, it is seen that the variation should always be by two—I, III, V, VII or II, IV, VI—and facts bear this out; mercury, iron, and copper being the only exceptions in the table just given. The irregular variations of these will be explained later.

Having considered the subject of chemistry from the standpoint of the individual elements, we can now take up the subject, grouping of elements, thus entering into the domain of chemical compounds.

We have spoken of molecules as the smallest particles of matter existing in a free state, and we mentioned among others the molecule of oxygen. Such an elemental molecule is called a *simple* molecule, in distinction to *compound* molecules; those containing at least two

different kinds of elements. Thus, while O_2 , S_2 , or P_4 are examples of simple molecules, $NaCl$, K_2SO_4 , and $KNaC_4H_4O_6$ are types of compound molecules; and while the simple molecules contain only a small number of atoms,—usually two,—the number of atoms in a compound molecule are practically unlimited; one complex organic body, hemoglobin, containing over 2000 atoms to the molecule, it is claimed.

Taking up the inorganic compound molecules, we first divide them into two classes—the binary and the ternary compounds.

Binary compounds are those containing two different kinds of elements. Thus, $NaCl$, KBr , CaO , and Cl_2O_3 are examples of binary compounds.

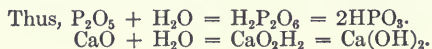
The naming of binary compounds is simple. We give the name of the positive element first; then the name of the negative element, changing its termination to “ide.” Making this clear by illustration, we will take $NaCl$ as the first example. This consists of sodium and chlorine. The table on p. 367 shows that sodium is the positive element and chlorine the negative element of that compound. Accordingly, we call the body sodium *chloride*. Remember, as *very important*, that the termination “ide” is characteristic of binary compounds.

In writing binary compounds we place the symbol of the positive element first; then the symbol of the negative element, placing below and to the right of each symbol, in Arabic numerals, the respective number of atoms entering into the molecule. Thus $CaCl_2$ means 1 atom of calcium and 2 atoms of chlorine, making a molecule of calcium chloride.

Before leaving binary compounds, mention must be made of the four acids belonging to this group. These four are HCl (hydrochloric acid); HBr (hydrobromic acid); HI (hydriodic acid); and HFl (hydrofluoric acid); and they are called the halogen or haloid acids. As acids will come in for discussion in Chapter XXIV, these halogen acids will be left for that time. They must be noted here, however, if for no other reason than as exceptions to the nomenclature—to the naming—of binary compounds.

Ternary compounds are those inorganic bodies containing three or more different kinds of elements. The word “ternary,” coming from a Latin root meaning “three,” would seem to exclude any compounds other than those containing three kinds of elements. However, as there are comparatively few definite inorganic compounds containing more than three different elements, these few are usually included and discussed with the ternary compounds. The naming of ternary compounds is a matter of some complexity, and before discussing this phase of the question it is necessary to explain the formation of ternary compounds.

All ternary compounds are derived indirectly from binary compounds, the oxides, and it is therefore necessary to discuss these important bodies. If we take the various oxides and combine them with water, we get, according as the distinctive element is positive or negative, an acid or a base, as shown by the two following equations:



By the term "equation," given above, we mean the representation by means of symbolic formulas of how a chemical reaction occurs.

Thus, the first of the two equations given above means that one molecule of phosphoric oxide (P_2O_5) combines with one molecule of water (H_2O) to make two molecules of metaphosphoric acid ($2HPO_3$).

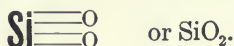
The second means that one molecule of calcium oxide (CaO) combines with one molecule of water (H_2O) to make one molecule of the base, calcium hydroxide ($Ca(OH)_2$).

Details of the writing and the balancing of such equations will be given later (p. 421).

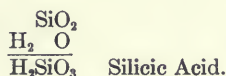
In discussing this matter we will consider, first, the acids and then the bases.

Formation of the Acid Formula.—Going back to the table on p. 367, running down the line of negative elements we find silicon with the valence four.

One atom of silicon with valence four can take up two atoms of oxygen with valence two, as shown in the following graphic formula:



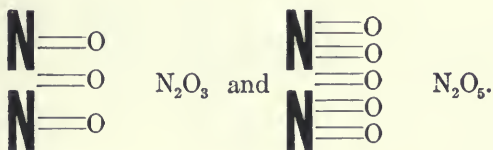
This body is called silicon oxide or silicic oxide. When this oxide combines with water we get:



In another form, $\text{SiO}_2 + \text{H}_2\text{O} = \text{H}_2\text{SiO}_3$.

It will be noticed that in this oxide and the derived acid the word silicon is changed to *silicic*.

Suppose we consider the two oxides of nitrogen which combine with water. Looking at the table on p. 367, we find the valence of nitrogen three and five; therefore the oxides must be



The linkage of the nitrogen and of the oxygen in these two cases occurs in a manner precisely the same as the linkage between oxygen and chlorine of valence three and five, as shown on p. 365. In naming these two oxides of nitrogen both cannot be *nitric* oxide, so we apply that name to the one in which nitrogen shows the highest valence, calling the other, the one in which nitrogen shows the valence three, *nitrous* oxide.

Adding water to these, we get the corresponding acids; thus—

- (a) N_2O_3 (*nitrous* oxide) + $\text{H}_2\text{O} = \text{H}_2\text{N}_2\text{O}_4 = 2\text{HNO}_2$ (*nitrous* acid).
 (b) N_2O_5 (*nitric* oxide) + $\text{H}_2\text{O} = \text{H}_2\text{N}_2\text{O}_6 = 2\text{HNO}_3$ (*nitric* acid).

These two equations can be expressed in words as follows:

(a) One molecule of nitrous oxide plus one molecule of water gives two molecules of nitrous acid.

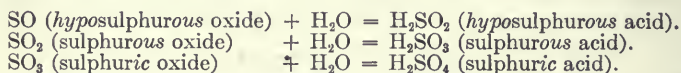
(b) One molecule of nitric oxide plus one molecule of water gives two molecules of nitric acid.

In other words, with these oxides of nitrogen the molecule of the oxide does not blend with the molecule of water to make one molecule of the acid, as was the case in the formula of silicic acid, but *two* molecules result from the combination.

As the symbolic formula expresses the contents of a molecule, the formula of nitrous acid is HNO_2 and the formula of nitric acid is HNO_3 .

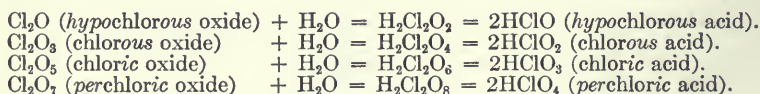
Looking up sulphur, we find it has three valences, and, therefore, yields three oxides, SO , SO_2 , and SO_3 . SO_3 we call *sulphuric* oxide; SO_2 , *sulphurous* oxide; and SO we call *hyposulphurous* oxide, the prefix "*hypo*" meaning "*under*."

Let us see how these three oxides form acids.



So when we have three acids from one negative element, the name of the negative element in which that element acts with the highest valence is given the termination "*ic*"; the termination in the one in which the element acts with the next lower valence is "*ous*"; while the one in which the element acts with the lowest valence is given the prefix "*hypo*" and the termination "*ous*."

As noted on p. 365, four oxides of chlorine yielding acids are known: Cl_2O , Cl_2O_3 , Cl_2O_5 , and Cl_2O_7 . In naming these, the one with the lowest valence of chlorine is called "*hypochlorous*"; the next, "*chlorous*"; and the next, "*chloric*," just as in the preceding. This leaves the fourth to be named, and it is given the prefix "*per*" and the termination "*ic*"—"perchloric." These four oxides yield four acids, as follows:



This completes the theoretic formation and naming of acids; the summary of nomenclature being:

If there is one oxyacid from an element it has the termination "*ic*."

If there are two, the higher in valence is "*ic*."

the lower in valence is "*ous*."

If there are three, the highest in valence is "*ic*."

the middle in valence is "*ous*."

the lowest in valence is "*hypo—ous*."

If there are four, the lowest in valence is "*hypo—ous*."

the next higher in valence is "*ous*."

the next higher in valence is "*ic*."

the highest in valence is "*per—ic*."

Remember, this method of nomenclature applies only to oxyacids—those acids which contain oxygen.

The four haloid acids have their specific names—hydrochloric, hydrobromic, etc., and do not enter into this category. Do not confuse *hydrochloric* with *chloric* acid. Hydrochloric acid is a binary compound, HCl, while chloric acid is ternary—an oxyacid—HClO₃.

It may be well to emphasize that the above explanation of the formation of acids is given merely with the view of teaching the student the writing of formulas, and it is necessary to warn the reader against assuming the conclusion that the practical manufacture of the chemicals whose formulas are thus worked out of necessity follows the same line here suggested. While it is true that sulphuric acid is made by treating sulphuric oxide with water, silicic acid cannot be formed by direct combination of silicic oxide (sand) and water. Again, it might be mentioned that frequently some of the compounds spoken of familiarly when discussing formula writing may be too unstable to exist. This is true of several oxides of chlorine and of iodine.

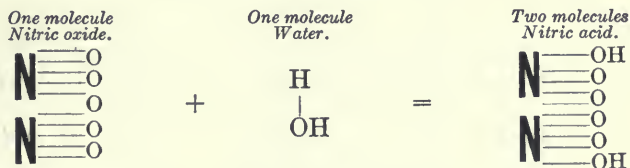
In working out the theoretic formation of the acids given above, the graphic formula of the oxide has been given, followed by the mere statement that the acid is formed by adding water to the oxide. The question that might arise is, How is it possible to add a molecule of water to a saturated compound? In the group SO₃ each of the six bonds of the sulphur are taken up by oxygen, so where can the two atoms of hydrogen and one of oxygen be attached?

The answer is very simple, for when water (H₂O) is brought in contact with sulphuric oxide (SO₃), the latter arranges with the water to the new groupings here shown:



the water breaking into H and OH; one of the O atoms of SO₃ breaking loose from the double union with sulphur and uniting with the free H from the water (forming OH), while the OH group from the water attaches itself to the free sulphur bond, and we get the compound SO₂(OH)₂—H₂SO₄, sulphuric acid—containing two hydroxyls (the radical mentioned on p. 630).

It might be interesting to express graphically the transition of one molecule of nitric oxide into two molecules of nitric acid, as described above.



It will be seen from this that the graphic formula of nitric acid is

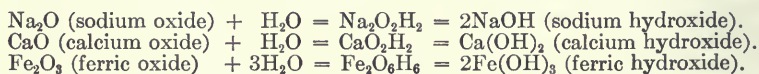


that is, to the atom of nitrogen there are attached two atoms of oxygen and one hydroxyl group.

Formation of the Formula of Bases.—Bases, with the exception of ammonia and its organic derivatives, which we will discuss in due course of time, represent the oxygen and hydrogen compounds of a positive element, and can be derived in exactly the same way as the acids have been from the negative elements—by adding water to the oxides; only in the case of bases the process is much simpler.

In glancing over the positive elements on p. 367, it will be noticed that all these elements, save mercury, iron, and copper, usually exhibit but one valence, and, accordingly, each element usually yields but one base. The nomenclature is much simplified by this fact, bases being named by placing the name of the characteristic element first, followed by the word "hydroxide."

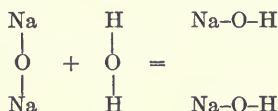
Let us summarize by a few general examples:



In the above equations the end formula, whenever possible, has been reduced to its simplest form. Thus, $\text{Na}_2\text{O}_2\text{H}_2$, like $\text{H}_2\text{N}_2\text{O}_4$ of p. 369, can be divided by two, and in truth the molecule of sodium hydroxide is NaOH and not $\text{Na}_2\text{O}_2\text{H}_2$. Again, note that a large figure placed at the commencement of a formula multiplies every element following it by that number; thus 2NaOH means $\text{Na}_2\text{O}_2\text{H}_2$; 2HNO_2 is the same as $\text{H}_2\text{N}_2\text{O}_4$ ($\text{O}_2 \times 2$ equals O_4).

It will be seen in the case of calcium hydroxide that CaO_2H_2 , containing as it does but one atom of calcium, constitutes one molecule. In such cases, however, it is considered neater to inclose the oxygen and hydrogen in brackets, with the number of atoms outside the brackets. Thus, CaO_2H_2 is usually expressed as Ca(OH)_2 . A small figure outside the brackets multiplies each element within the brackets, even as a large figure in front of the formula multiplies every element following it.

Graphically, these hydroxides may be expressed with equal ease as hydroxyl derivatives, each bond of metal taking one OH group, thus:



It is customary to make a slight difference in the writing of acids and bases. In writing acids, we place the hydrogen atoms first, then the characteristic element, and finally the oxygen atoms. In bases, on the contrary, the characteristic element comes first, followed by the hydroxyl groups. This is a mere matter of form, the only reason why the two classes of bodies are so differentiated being because it affords a convenient method of distinguishing acid and basic formulas.

It is wise, however, not to get bound down to rigid rules. Different books have different ways of writing formulas; for instance, sulphuric acid can be written $\text{SO}_2(\text{OH})_2$ as well as H_2SO_4 .

Considerable mention has been made on preceding pages of acids and bases without giving any idea what these bodies really are. The theoretic consideration of them just given is very important,

but it does not represent real practical conditions. Acids and bases had first to be known before theories regarding them were evolved, and it is of greatest importance that they should be known when met with.

The following definitions should be carefully studied:

An *acid* is a substance which turns *blue* litmus-paper *red* and combines with bases to form salts.

A *base* is a substance which turns *red* litmus-paper *blue* and combines with acids to form salts. And, finally,

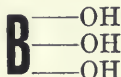
A *salt* is the product of the combination of an acid and a base.

Litmus-paper is an article of commerce made by dipping porous paper in a decoction of litmus, a sort of lichen. A drop of acid (always to be applied in diluted form) will make a red spot on blue litmus-paper, and likewise a drop of diluted alkali (as bases are called) will make a blue spot on red litmus-paper.

The three classes, acids, bases, and salts, cover the entire domain of ternary compounds. Before speaking of salts, mention must be made of another point concerning acids. We have just seen that sulphuric acid may be written—



In other words, as a sulphur atom with two oxygen atoms and two hydroxyl groups attached. If we look up the formula of boric acid, we find it to be H_3BO_3 or, graphically,

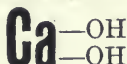


In this, each bond of the characteristic atom is taken up by a hydroxyl, and such an acid is called an "ortho" acid. Almost all bases are "ortho," having as many hydroxyls as bonds of the characteristic element, and thus we find—

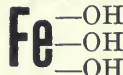
Sodium hydroxide,



Calcium hydroxide,



Ferric hydroxide,



but with acids it is different, and of the oxyacids, *boric acid* is the only official ortho acid.

There is an official acid called orthophosphoric acid (H_3PO_4), but it is not a true ortho-acid in the sense expressed above. Its graphic formula is—



while true ortho-phosphoric acid should be

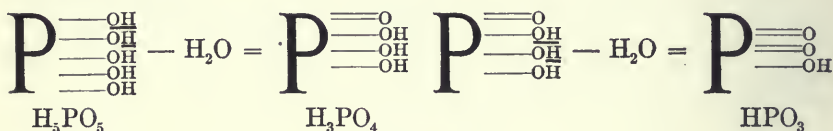


Acids not having all the bonds of the characteristic element taken up by hydroxyl groups are called *meta*-acids, and, accordingly, H_3PO_4 should be classed among the *meta*-acids.

The original idea of *ortho*- and *meta*- combinations was that when two kinds of salts of the same acid were known, the one containing the most metal was called the *ortho*-salt, while the other was called the *meta*-salt. Thus, the sodium salt of H_3PO_4 was called orthophosphate of soda, while the sodium salt of HPO_3 was called metaphosphate of soda.

The difference between the three phosphoric acids, H_5PO_5 , H_3PO_4 , and HPO_3 , is simply a loss of water.

It appears that usually the characteristic element cannot carry many hydroxyl groups, and these are parted with by the throwing off of water, as represented in the following graphic sketch, in each formula of which the hydrogen and oxygen, thrown off as water, are underscored:



SALTS

Salts are solids, neutral to test-paper, affecting neither the red nor the blue of litmus. Of course, there are exceptions; but this much can be usually stated regarding them: As to the naming of a salt, we place first the name of the metal (the base; the positive element), then the name of the acid, the termination of which is changed to "ate," if we are dealing with an "ic" acid; and to "ite," if it comes from an "ous" acid. This is shown clearly in the names of the oxyacids of chlorine.

Hypochlorous acid gives hypochlorites.

Chlorous acid gives chlorites.

Chloric acid gives chlorates.

Perchloric acid gives perchlorates.

The salts of the halogen acids do not come under this method of nomenclature. Such salts are binary compounds, and are given the termination "ide"; thus, hydrochloric acid yields chlorides, hydrobromic acid yields bromides, hydriodic acid yields iodides. The alkaloidal salts of these acids are, however, called hydrochlorates, hydrobromates, hydriodates respectively.

The formation of salts is governed by valence, just as is the origin of all other compounds, and to write the formulas of salts intelligently it is essential to know the valence expressed in the table on p. 367.

We have given as the definition of valence "the relative combining power of elements." Valence, however, means more than this, and when we say that the valence of sodium is one, we not only mean that an atom of it can combine with one atom of chlorine to form a molecule of sodium chloride, but also (what is the same thing expressed differently) one atom of it can *replace* one atom of hydrogen in hydrochloric acid (HCl) to form the molecule of sodium chloride. So valence not only means relative combining power, but also the relative power of a metal to replace the hydrogen of an acid.

A few examples may perhaps better explain this.

Let us take sodium, with the valence one. One atom can replace one atom of hydrogen in an acid.

Nitric acid, HNO_3 , has one atom of hydrogen; this can be replaced by an atom of sodium, when we get NaNO_3 , sodium nitrate. Sulphuric acid, H_2SO_4 , has two atoms of replaceable hydrogen; therefore, two atoms of sodium are needed, when we get Na_2SO_4 , sodium sulphate.

Now for the combinations of *calcium*, valence two, which means that one atom of calcium can replace two atoms of hydrogen in an acid.

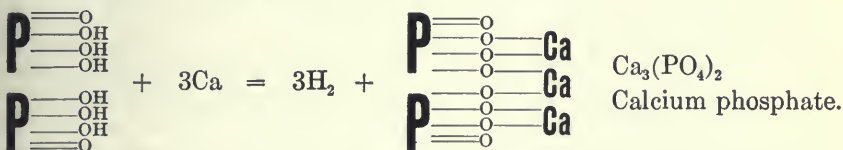
Nitric acid, HNO_3 , has one atom of hydrogen; calcium requires two; accordingly, we take one atom of calcium and two molecules HNO_3 , thus:



replacing the two hydrogen by one Ca, forming $\text{Ca}(\text{NO}_3)_2$, calcium nitrate.¹

Sulphuric acid, H_2SO_4 , has two atoms of hydrogen. These can be replaced by one atom of calcium, when we get CaSO_4 , calcium sulphate.

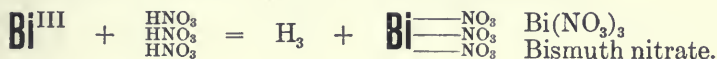
Phosphoric acid, H_3PO_4 , has three replaceable atoms of hydrogen; calcium requires two. This complicates matters and we had better write the equation graphically:



the reaction occurring between three atoms of calcium and two molecules of phosphoric acid, the six hydrogen atoms of which are replaced by the three calcium atoms.

As the last example, let us take bismuth, valence three; one atom of bismuth being able to replace three atoms of hydrogen in an acid.

Nitric acid, HNO_3 , has one hydrogen; so to combine with bismuth three molecules of nitric acid are necessary, thus:



Sulphuric acid, H_2SO_4 , has two atoms of hydrogen; bismuth requires three; therefore combination occurs between three molecules of the acid and two atoms of bismuth, thus:



¹ In this and other replacing equations found in this chapter, the liberation of hydrogen need not be taken too literally. Metallic calcium plus the appropriate acid may liberate hydrogen, but the reaction is too violent to be practical. In general manufacturing the oxide or carbonate of the first and second group of metals is usually employed.

Phosphoric acid, H_3PO_4 , has three atoms of hydrogen; bismuth requires three, so the combination will be:



To sum up the important study of the writing of formulas of salts:

Halogen Salts.—These follow the general rule for binary compounds. Remember that all official compounds the name of whose negative element ends in “ide” are binary, and then follow the general rule for binary formulas given on p. 368; the process being merely a matter of relative valence. As to writing binary compounds, look up the valence of the positive and negative elements of the compound, and combine these in just such proportions that each will furnish the same number of bonds; viz.:

Valences similar,	the elemental union is atom for atom.		
Valences II and III,	“	“	3 atoms of II to 2 atoms of III.
Valences II and IV,	“	“	2 atoms of II to 1 atom of IV.
Valences II and V,	“	“	5 atoms of II to 2 atoms of V.
Valences II and VI,	“	“	3 atoms of II to 1 atom of VI.
Valences II and VII,	“	“	7 atoms of II to 2 atoms of VII.

Combinations of the valence I with the valence II to VII are so simple that they need not be given. Until the student becomes expert, it is better to work out formulas by the graphic means described on p. 369.

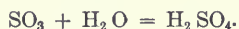
In writing out the empirical formulas of the binary compounds, remember that the symbol of the positive element comes first, then that of the negative, with the number of atoms of each below and to the right of the respective symbols.

Ternary Salts.—In writing a formula of a ternary salt, we first must know the formula of the acid from which it is derived, and this is worked out from the formula of the oxide, which in turn is figured by valence—by the regular rule for the writing of binary formulas. An example can best illustrate this.

We want to know the formula of sodium sulphate and do not know either the formula of sulphuric acid nor of sulphuric oxide. Referring to table of elements on p. 367, we find the valence of sulphur is II, IV, and VI. We are working on sulphuric compounds, and the valence of sulphur in these is its highest, VI, and, graphically, we sketch the oxide.



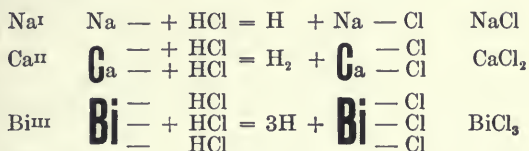
To make this into sulphuric acid, water is added, by the following reaction:



Thus, learning that the formula of sulphuric acid is H_2SO_4 , and remembering that each atom of hydrogen removed from the acid in making a salt calls for one bond of the replacing metal, we now look at the *replacing capacity*—the valence—of the metal. We find that sodium has the valence one—that one atom of it can replace but one

atom of hydrogen in an acid. As sulphuric acid has two atoms of hydrogen, to replace these, two atoms of sodium will be needed, and by this reasoning we learn that the formula of sodium sulphate is Na_2SO_4 . All this was fully explained on p. 375.

If deemed preferable, the haloid salts—the salts of hydrochloric, hydrobromic, hydriodic, and hydrofluoric acids—may be worked out by this idea of replacement of hydrogen in an acid by a metal, as the following sketching of the formulas of chlorides will show:



This explanation can be condensed into the following table of formula writing. If we want the formula of a ternary salt—say sodium sulphate—it can be worked out by answering the following questions:

QUESTION.

ANSWER.

- | | |
|---|--|
| 1. Of what acid is the compound a salt? | Applied to the type, sodium sulphate.
A <i>sulphate</i> is a salt of sulphuric acid, "ic" acids giving "ate" salts. |
| 2. From what oxide is this acid derived? | From sulphuric oxide, the name of the oxide being always that of the acid.
Sulphuric acid must come from the element sulphur. |
| 3. What is the characteristic element in this oxide? | II, IV, and VI, as shown on p. 367. |
| 4. What is the valence of this element? | "ic" oxide means that oxide showing the highest valence of the element, when that element has three or less valences. Hence in sulphuric oxide sulphur must have the valence VI. |
| 5. What is the valence of this element in the oxide above mentioned? | One atom of sulphur with valence VI takes three atoms of oxygen (valence II) for saturation, thus: |
| 6. What is the graphic formula of this oxide? | $ \begin{array}{c} \text{S} \begin{array}{l} \text{---} \text{O} \\ \text{---} \text{O} \\ \text{---} \text{O} \end{array} \\ \text{---} \text{O} \end{array} - \text{SO}_3. $ |
| 7. How is the acid derived from this oxide? | By addition of water, thus, $\text{SO}_3 + \text{H}_2\text{O} = \text{H}_2\text{SO}_4$. |
| 8. What, then, is the formula of the acid? | H_2SO_4 . |
| 9. What is the valence of the metal in the salt? | The table on p. 367 says sodium has the valence I. |
| 10. What does this mean? | That one atom of sodium can replace one atom of hydrogen in an acid. |
| 11. How many atoms of replaceable hydrogen are there in the acid in question? | In H_2SO_4 there are two atoms of replaceable hydrogen. |
| 12. Then how many atoms of the metal are needed to make a molecule of the salt in question? | The two atoms of hydrogen in H_2SO_4 need two atoms of sodium to replace them. |
| 13. What, then, will be the formula of the salt? | Its formula will be Na_2SO_4 . |

By such reasoning, with the aid of the table on p. 367, practically every formula of a ternary salt can be worked out.

The salts produced as just described—by replacing all the hydrogen of an acid by a metal—are called *normal salts*.

Thus, when the two atoms of hydrogen in sulphuric acid (H_2SO_4) are replaced by two atoms of sodium, we obtain Na_2SO_4 —normal sodium sulphate.

It is possible, however, to replace only one atom of hydrogen in the above acid by one atom of a monad metal, and if sodium is the metal, we obtain NaHSO_4 (sodium bisulphate), one hydrogen still remaining in the compound. This atom of hydrogen still exerts its acid properties, and such salts usually turn blue litmus red, and are, therefore, called *acid salts*. Why these salts are given the prefix “bi,” as *bisulphate*, *bitartrates*, etc., will be explained later.

On treating these *acid salts*, such as potassium bitartrate ($\text{KHC}_4\text{H}_4\text{O}_6$), with the hydroxide or carbonate of another metal (and be it here said that in practice the metal itself is rarely used in making these salts, in many cases it being too dangerous a procedure), the other metal replaces the remaining atom of replaceable hydrogen, thereby forming a *double salt*.

Thus, replacing the hydrogen of $\text{KHC}_4\text{H}_4\text{O}_6$ by an atom of sodium, we obtain potassium and sodium tartrate, ($\text{KNaC}_4\text{H}_4\text{O}_6$), or Rochelle salt.

In due course of time (p. 633) it will be explained why only part of the hydrogen of an organic acid is replaceable by a metal.

Besides the normal, acid, and double salts just mentioned, there is a class of salts called *basic salts*. In the acid salts there is pre-dominance of the acid portion; in the basic salts there is a deficiency of such. These basic salts will be more thoroughly considered later. (See p. 561.) So here suffice it to state that most of the “sub” preparations, as bismuth *subnitrate*, iron *subsulphate*, etc., are basic salts.

CHEMICAL ARITHMETIC

The pharmaceutical application of atomic and molecular weights described on pp. 362 and 363 are manifold.

Thus, through molecular weights we are able to *estimate the amount of water of crystallization* in a chemical (p. 147).

For instance, let us figure out the percentage of water of crystallization in the formerly official sodium carbonate.

The formula of this body is $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, which means a molecular weight of 286, as the following reckoning will show:

Atomic weight of Na	is 23	$\text{Na}_2 = 2 \times 23 =$	46
Atomic weight of C	“ 12	$\text{C} = 1 \times 12 =$	12
Atomic weight of O	“ 16	$\text{O}_3 = 3 \times 16 =$	48
Atomic weight of H	“ 1	$10\text{H}_2 = 20 \times 1 =$	20
Atomic weight of O	“ 16	$10\text{O} = 10 \times 16 =$	160
			286

The molecular weight of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ is 286, and of this, 180 parts ($20 + 160$) represent the ten molecules of water of crystallization. What per cent. of 286 is 180?

This is easily $\frac{180}{286}$ of 100 per cent., or 63 per cent., and we thus learn that 63 per cent. of crystalline sodium carbonate is water.

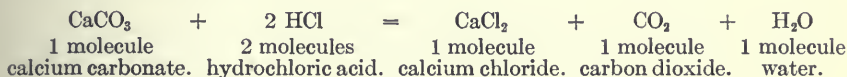
Again, the knowledge of molecular weights is imperative in estimating the *proportional weight of substances entering into a chemical reaction*.

For instance, let us calculate the amount of calcium carbonate and hydrochloric acid needed to make calcium chloride.

Such a problem is solved as follows:

We first figure out what the reaction would be, and express same by the appropriate equation.

In this case it is:

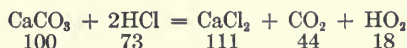


What are the molecular weights of these several bodies?

<u>CaCO₃.</u>		<u>HCl.</u>	
At. wt. of Ca = 40.	Ca = 1 × 40 = 40	At. wt. of H = 1.	H = 1 × 1 = 1
At. wt. of C = 12.	C = 1 × 12 = 12	At. wt. of Cl = 35.5.	Cl = 1 × 35.5 = 35.5
At. wt. of O ₃ = 16.	O ₃ = 3 × 16 = 48	Mol. wt. of HCl = 36.5	
Mol. wt. CaCO ₃ = 100		Hence 2 molecules HCl = 73.0	

<u>CaCl₂</u>		<u>CO₂</u>		<u>H₂O</u>	
Ca = 1 × 40 = 40		C = 1 × 12 = 12		H ₂ = 2 × 1 = 2	
Cl ₂ = 2 × 35.5 = 71		O ₂ = 2 × 16 = 32		O = 1 × 16 = 16	
Mol. wt. CaCl ₂ = 111		Mol. wt. CO ₂ = 44		Mol. wt. H ₂ O = 18	

To write equation again:



This summed up means:

100 Gm. absolute calcium carbonate	Give 111 Gm. calcium chloride.
and 73 " " hydrochloric acid	44 " carbon dioxide.
173 Gm.	18 " water.
	173 Gm.

Observe that we obtain exactly the same number of grammes of new substances as the total amount of material with which we started in. In other words, in chemical reactions matter is not lost, but merely rearranged. (Law of conservation of matter.)

It is scarcely necessary to say that the amounts given in the above equation are merely proportional, that in place of 100 grammes and 73 grammes given above, we could put 100 pounds and 73 pounds; 100 ounces and 73 ounces; or 100 tons and 73 tons. All that is essential is that quantities of calcium carbonate and hydrochloric acid be in the proportion of 100 to 73.

Let us carry out several modifications of this idea:

(a) Suppose the example was: "How much calcium chloride can be made from 1 pound of calcium carbonate?"

From above equation we see that 100 pounds calcium carbonate give 111 pounds calcium chloride. We therefore reason thus:

If 100 pounds CaCO_3 give 111 pounds CaCl_2 , then 1 pound CaCO_3 will give $\frac{1}{111}$ of 111 pounds CaCl_2 , or 1.11 pounds CaCl_2 .

The same reasoning expressed in proportion reads:

$$\begin{array}{ccccccc} 100 & : & 111 & :: & 1 & : & \times \\ & & \times & = & \frac{111 \times 1}{100} & & \text{equals 1.11.} \end{array}$$

(b) Suppose the question was reversed, and read: "How many ounces of calcium carbonate and of absolute hydrochloric acid are needed to make 60 ounces of calcium chloride?"

Since this question asks quantities of the two ingredients, it is in reality two examples, and we must solve each separately.

From the above equation we learn:

100 ounces calcium carbonate and 73 ounces absolute hydrochloric acid make 111 ounces calcium chloride.

Applying this truth to the case under consideration, we reason—

If to make 111 ounces CaCl_2 we need 100 ounces CaCO_3 , then to make 60 ounces CaCl_2 we will need $\frac{60}{111}$ times 100 ounces CaCO_3 ,

$$\begin{array}{l} \text{or } \frac{6000}{111} \text{ ounces } \text{CaCO}_3, \\ \text{or } 54+ \text{ ounces } \text{CaCO}_3. \end{array}$$

Or expressed in proportion:

$$\begin{array}{ccccccc} 111 & : & 100 & :: & 60 & : & \times \\ & & \times & = & \frac{100 \times 60}{111} & & \text{equals } 54+. \end{array}$$

The second part of the example is solved by similar reasoning, viz.:

If to make 111 ounces CaCl_2 we need 73 ounces absolute HCl , then to make 60 ounces CaCl_2 we need $\frac{60}{111}$ times 73 ounces HCl , or $\frac{4380}{111}$ ounces HCl , or 39.46 ounces absolute HCl .

Or expressed in proportion:

$$\begin{array}{ccccccc} 111 & : & 100 & :: & 73 & : & \times \\ & & \times & = & \frac{100 \times 73}{111} & & \text{equals 39.46.} \end{array}$$

(c) The figures just given suggest a problem of simple proportion which can be best considered here.

It is seen that the answer just given is 39.46 ounces absolute (100 per cent.) hydrochloric acid. Now, 100 per cent. hydrochloric acid is a gas, and what we commonly call hydrochloric acid is a solution of this gas in water, the hydrochloric acid directed by the pharmacopœia being a solution containing 31.9 per cent. by weight of the gas (p. 412).

Now, suppose the above example asked: "How much hydrochloric acid U. S. P. is needed to make 60 ounces calcium chloride?"

We have just seen that to make 60 ounces CaCl_2 39.46 ounces 100 per cent. HCl is required. Now, if we use the weaker 31.9 per cent. acid, it will require a proportionally greater quantity, and indeed $\frac{100}{31.9} \times 39.46$ ounces, or $\frac{3946}{319}$ ounces, or 123.7 ounces of 31.9 per cent. hydrochloric acid.

The amount is reasoned out as follows:

If it takes 39.46 ounces of the 100 per cent., then it will take 100 times as much of the 1 per cent. (39.46 ounces of 1 per cent.) and $\frac{1}{31.9}$ of the last amount (exactly $\frac{100}{319}$), or $\frac{3946}{319}$, or 123.7 ounces.

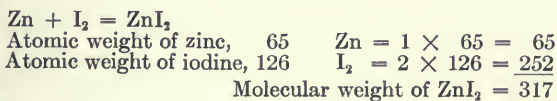
Or expressed by proportion:

$$\begin{array}{rclcl} 31.9 & : & 100 & :: & 39.46 & : & \times \\ & & \times & = & \frac{100 \times 39.46}{31.9} & \text{equals} & 123.7. \end{array}$$

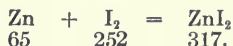
(d) In the same way we can estimate the amounts of elements combining to form a compound.

Suppose the question is: "How many grammes of zinc and of iodine are needed to make 100 grammes of zinc iodide?"

The question in this case runs as follows:



Rewriting the equation:



This means:

To make 317 Gm. ZnI_2 we need 65 Gm. of zinc and 252 Gm. of iodine. Then to make 100 Gm. ZnI_2 we need $\frac{100}{317}$ times 65 Gm. of zinc and $\frac{100}{317}$ times 252 Gm. iodine, or 20.5 Gm. of zinc and 79.4 Gm. of iodine.

Or by proportion:

$$\text{As to zinc, } 317 : 65 :: 100 : \times. \quad \times = \frac{65 \times 100}{317} = 20.5.$$

$$\text{As to iodine, } 317 : 252 :: 100 : \times. \quad \times = \frac{252 \times 100}{317} = 79.4.$$

Lastly, the knowledge of molecular weights is essential in quantitative analysis, both gravimetric and volumetric. This phase of the question will be left until Chemical Analysis is considered in Chapter LIV.

The writing and balancing of chemical equations, such as those given above, will be explained later (p. 421).

CHAPTER XXIII

THE NEGATIVE ELEMENTS AND THEIR COMPOUNDS

ON p. 366 we learned that the elements were roughly divided into the two classes, the negative elements and the positive elements, according to their behavior when submitted to electrolytic action. We have also seen that the hydroxides of the negative elements are called acids, the hydroxides of the positive elements are called bases, while the union of the acids and bases results in a class of compounds called salts.

The salts, along with the bases, will be considered under the metal they contain, and the acids will be considered in the next chapter, as it is advisable, before proceeding to a consideration of the official acids, to study the negative elements from which the important acids are derived, as well as those recognized by the pharmacopœia.

The negative elements are also called the non-metallic elements, or metalloids. The non-metals rarely possess a metallic appearance, are usually poor conductors of electricity, and combine with hydrogen to form gaseous bodies.

A list of the negative elements of importance in pharmacy is found on p. 367, while the following table shows those recognized by the pharmacopœia either as the free element or in combination other than in acids or in salts. In this list the negative metals, arsenic and antimony, are not mentioned, since their analytic behavior groups them among the metals, and they can there be more profitably discussed.

Water
Distilled water } (H_2O).

Solution of hydrogen dioxide (containing 3 per cent. of H_2O_2).

The Halogens:

Compound solution of chlorine (containing about 0.4 per cent. chlorine).

Chlorinated lime (containing 35 per cent. available chlorine).

Solution of chlorinated soda (containing 2.4 per cent. available chlorine).

Bromine (Br).

Iodine (I):

Tincture of iodine (containing 7 per cent. iodine).

Compound solution of iodine (containing 5 per cent. iodine).

Ointment of iodine (containing 4 per cent. iodine).

Sublimed sulphur (S):

Washed sulphur.

Precipitated sulphur.

Sulphur iodide (S_2I_2).

Carbon disulphide (CS_2).

Phosphorus (P).

Pills of phosphorus ($\frac{1}{10}$ of 1 milligramme to a pill).

Wood-charcoal

Animal-charcoal

Purified charcoal

} (Carbon in condition of greater or less purity).

Neither *hydrogen* nor *oxygen* is recognized by the U. S. P., but both elements are worthy of notice at this place.

Hydrogen is made by treatment of zinc or other metals with sulphuric acid, and the details of the manufacture will be given later (see p. 519). It is a colorless, odorless, and tasteless gas, and is interesting as being the lightest known substance. By reason of this

fact it is chosen by chemists as the standard of atomic weight and of a large number of other chemical and physical measurements. *Oxygen* can be made by heating potassium chlorate alone, or preferably by heating a mixture of potassium chlorate and manganese dioxide, the addition of the latter substance being to prevent too rapid an evolution of the gas. Oxygen is a colorless, odorless, and tasteless gas, which is non-inflammable, but one of the best supporters of combustion. Its use in medicine is becoming more extended day by day, it being used to aid respiration in extreme cases where the lungs refuse to absorb ordinary air. For such emergency cases a good quality of oxygen is absolutely necessary, and it behooves the pharmacist to arrange to procure the gas with little loss of time. The manufacture of the gas for such purposes by the pharmacist is rarely practicable, and, moreover, scarcely necessary, inasmuch as the various firms in different portions of the country will furnish an excellent quality of oxygen in compressed (liquid) form at a comparatively low figure. Yet it may here be stated that the writer knows of a pharmacist in a small city who developed quite a business and materially enhanced his reputation by furnishing chemically pure oxygen in rubber bags for just such emergency cases. While neither hydrogen nor oxygen is recognized by the pharmacopœia, two compounds of these elements are official, *water* and *hydrogen dioxide solution*.

AQUA. Water.



Potable Water in its purest obtainable state.

A colorless, limpid liquid, without odor or taste at ordinary temperatures, and odorless when heated.

Water should be perfectly neutral to litmus-paper. 1000 Cc. when concentrated by evaporation to 20 Cc., should not respond to the Time-Limit Test for *heavy metals* (see p. 979).

On evaporating 1000 Cc. of Water on a water-bath, it should not leave a residue weighing more than 0.5 Gm. (limit of *soluble salts*), and this residue, when ignited, should not carbonize, nor evolve *ammoniacal* or *acid* vapors.

If 200 Cc. of Water be acidulated with hydrochloric acid, heated to boiling, and 0.5 Cc. of barium chloride T.S. be added, the liquid, when cooled and filtered, should give no further precipitate on the addition of a few drops of barium chloride T.S., even on standing (limit of *sulphates*).

If 200 Cc. of Water be acidulated with nitric acid, heated to boiling, and 0.5 Cc. of tenth-normal silver nitrate V.S. be added, the liquid, when cooled and filtered, should not be affected by the subsequent addition of a few drops of silver nitrate T.S. (limit of *chlorides*).

If 10 Cc. of Water mixed with a few drops of diphenylamine T.S. be carefully poured upon about 3 Cc. of sulphuric acid, free from nitrous compounds, contained in a test-tube, so as to form a separate layer, no blue color should be formed at the line of contact of the two liquids (limit of *nitrates*).

If to 50 Cc. of Water contained in a glass cylinder 2 Cc. each of sulphanilic acid T.S. and naphthylamine acetate T.S. are added, and the solution well mixed, no distinct pink coloration should appear within five minutes, if the cylinder be placed upon a white surface and viewed from above (limit of *nitrites*).

If to 50 Cc. of Water contained in a glass cylinder 2 Cc. of alkaline mercuric potassium iodide T.S. (Nessler's Reagent) be added and thoroughly mixed, no yellow or brownish tint should be produced immediately; the cylinder should be placed upon a white surface and viewed from above (limit of *ammonia*).

On heating to boiling 100 Cc. of Water, acidulated with 10 Cc. of diluted sulphuric acid, and subsequently adding 0.4 Cc. of tenth-normal potassium permanganate V.S., the pink color of the liquid should not be completely destroyed after it has been boiled for ten minutes (limit of *organic* or *other oxidizable substances*).

reaction into the insoluble manganese dioxide. If the water is free from organic matter, no such chemical change occurs, hence the rose tint will remain. This, however, is not an absolutely fair hygienic test, as the presence of innocent organic substances—say, even sugar—produces a similar discharge of the rose tint. Accordingly, the permanganate is of value chiefly from the negative side, that is, water which does not discharge the permanganate tint can be considered free from organic matter, but, on the other hand, the discharging of the color does not necessarily mean the presence of pernicious organic matter. Therefore in examining water the permanganate test should always be considered in conjunction with three other tests recognized by the pharmacopœia—the diphenylamine test for nitrates, the sulphanilic acid test for nitrites, and the Nessler test for ammonia. In water analysis the last is the most important chemical test for sewage contamination. In the thorough application of this test the water is distilled from an alkaline solution and then from alkaline permanganate solution, whereby any nitrogenous matter present in the water is converted into ammonia, which is detected by treating the distillate with a drop or so of Nessler's reagent. (See p. 484.) This reagent will produce either a yellow coloration or a brown precipitate, according to the amount of ammonia present, and from the extent of coloration has been evolved a scheme for the quantitative estimation of the albuminoid ammonia present in the water. These chemical tests are of value merely in establishing the fact of sewage contamination, and it may here be said that the simple fact of such contamination would be of little danger were the sewage free from the numerous bacteria which cause disease. Of the bacteria causing mischief in potable water, the most dangerous is that of typhoid, which can be detected only by an expert bacteriologist, and that only indirectly, as the identification of the bacilli is so difficult that the so-called typhoid tests are usually based on the detection of the intestinal bacilli, such as *Bacillus coli communis*. In closing the subject mention may be made of the fact that the best method of water purification is by filtration through deep layers of sand. (See p. 132.) The pharmacopœia specifies that *aqua* should be potable water in its purest obtainable state, and such water, after having been proved potable, is permitted to be used by the pharmacist in most of his manufacturing. A water may be hygienically pure and yet contain salts, the presence of which, while nowise affecting the potability of the water, would seriously interfere with the use of such water in the manufacture of chemicals. For this reason the careful pharmacist usually prefers to use in all prescription work distilled water, the price of which is now so low that its general use is not an extravagance.

Distilled water is made, according to the pharmacopœia, by taking a thousand volumes of water, distilling it from a suitable apparatus, rejecting the first 100 volumes which come over, then collecting the subsequent 800 volumes. It will be seen by this process that the first 100 and the last 100 volumes of water are thrown away, the intermediate portion being the only part considered fit for use. The explanation of this procedure is found in the fact that ordinary water is usually impregnated with gaseous substances, such as carbon

dioxide and possibly ammonia, and that these gases will pass over with the first 100 volumes; hence this portion is thrown away. The same water contains also solid matter, various salts, and possibly fixed organic material, and this remains in the still, and the pharmacopœial provision against complete evaporation of the water from the residue is to prevent charring, with the subsequent evolution of empyreumatic matter.

The pharmacopœial tests for distilled water are much more severe than for ordinary water. For instance, while the permanganate test applied to ordinary water is that the tint should not be discharged after boiling 100 Cc. of water with 0.4 Cc. of decinormal permanganate solution for ten minutes, distilled water is directed to be mixed with 0.1 Cc. decinormal potassium permanganate solution (one-quarter the quantity), and it is further stated that this color should not be dis-

charged after boiling ten minutes, nor by afterward setting the vessel aside for ten hours. In the storing of distilled water more care should be taken than is usually observed. Tightly corking bottles of distilled water generally results in the development, in the water, of microscopic plants (*confervidæ*). Experience has shown that if the water is kept in bottles which are loosely stoppered with a pledget of cotton, such growths are not so frequent. At the prescription counter a capital way of handling the water is to place it in a half-gallon, wide-mouthed bottle on the shelf, six or seven feet above the floor. A syphon consisting of a bent glass tube with flexible rubber tube at the end, the rubber tube terminating with a tapering glass tube and provided with a pinch-cock, is placed in the water, the orifice of the bottle being closed with cotton. When water is desired, all that is necessary is to open the pinch cock and allow the water to syphon into the bottle or graduate, which can then be held on a level with the eye without the necessity of raising the heavy water receptacle (Fig. 215).

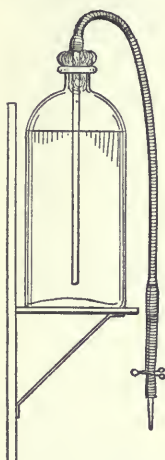


Fig. 215.—Distilled water-holder.

on a level with the eye without the necessity of raising the heavy water receptacle (Fig. 215).

AQUA HYDROGENII DIOXIDI. Solution of Hydrogen Dioxide.

A slightly acid, aqueous solution of Hydrogen Dioxide [$\text{H}_2\text{O}_2 = 33.76$], which should contain, when freshly prepared, about 3 per cent., by weight, of absolute Hydrogen Dioxide, corresponding to about 10 volumes of available oxygen. It should be kept in a cool place. Upon removing the stopper from the bottle not more than a slight pressure should be observed.

A colorless liquid, without odor, slightly acidulous to the taste, and producing a peculiar sensation and soapy froth in the mouth; liable to deteriorate upon keeping or protracted agitation. If the stopper in the bottle be replaced by a pledget of cotton, deterioration is retarded.

When exposed to the air at the ordinary temperature, or when heated on a water-bath at a temperature not exceeding 60°C . (140°F .), the solution loses chiefly water. When rapidly heated, it frequently decomposes suddenly.

On adding to 10 Cc. of water, in a test-tube, 1 drop of potassium chromate T.S., then 10 drops of diluted sulphuric acid, and pouring a few Cc. of ether on top, the subsequent addition of a few drops of Solution of Hydrogen Dioxide, even when considerably diluted, will cause a blue color to appear at the zone of contact of the two liquids. After shaking the ether-layer will separate with a blue color.

If to 25 Cc. of the Solution 5 Cc. of tenth-normal potassium hydroxide V.S. be added, and the mixture be evaporated to about 10 Cc., and 3 drops of phenolphthalein T.S. added, not less than 2.5 Cc. of tenth-normal sulphuric acid V.S. should be required to discharge the red color of the solution after continued boiling (limit of *free acids*).

If 20 Cc. of the Solution be evaporated to dryness upon a water-bath, and the drying completed at 120° C. (248° F.), not more than 0.03 Gm. of solid residue should remain (limit of *total solids*).

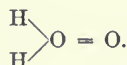
Tests for Identity.—See above.

Impurities.—Free acids, arsenic, heavy metals, hydrofluoric acid, and barium. Details of testing of these are given in Part V. For excess of solids and of free acid, see above.

Assay.—Details in Part V.

Remarks.—Hydrogen dioxide was discovered by Thenard in the year 1818, and is recognized by the pharmacopœia in the form of a 3 per cent. solution under the name of Aqua Hydrogenii Dioxidi.

The formula H_2O_2 has given rise to considerable discussion as to the structure of this body. At the first glance it seems to lead to the conclusion that either oxygen has the valence I or hydrogen has the valence II. An explanation, however, covering the usual valences of these two elements is that the body consists of two hydroxyl groups, viz., $HO-OH$. This, however, is combatted by several investigators, who claim that if such a linkage were true, the oxygen would be more tightly combined in this body than it is in water, while the reverse is true. Others claim that the graphic formula of hydrogen dioxide is



that is, it exhibits one atom of tetravalent oxygen; while still others think it represents water with an extra atom of oxygen very loosely attached thereto. In this connection it may be stated that practically all bodies, the formulas of which are not in harmony with the accepted ideas of valence, are usually of unstable character, and no better example of this need be given than the unstable hydrogen dioxide.

The official solution of hydrogen dioxide is prepared by treating barium dioxide with phosphoric acid. The usual method of manufacturing, however, is by substituting for the phosphoric acid the cheaper sulphuric acid. The equation of manufacture from sulphuric acid is as follows:



From this will be seen that the barium dioxide is converted into the insoluble barium sulphate, from which the solution of hydrogen dioxide can be removed by simple filtration.

The pharmacopœia provides an assay of hydrogen dioxide, based on the quantity of potassium permanganate solution required to oxidize the same.

The official solution is a clear, colorless liquid, having a slightly acidulous taste, producing a peculiar sensation and a soapy froth in the mouth. This peculiar sensation is due to its oxidizing action on the mucous membrane and its excretions. Its chemical and medical action is based entirely on the amount of oxygen that it gives up under such conditions, and it may be here said that the solution, giving off its oxygen on standing, deteriorates rapidly. Not only

this, but the evolution of oxygen in tightly closed bottles is sometimes so considerable as to cause an explosion. Hence it is advisable to avoid an overtight stopping of the bottle, unless the latter is very stout. Manufacturers preserve hydrogen dioxide solution by the addition of a small amount of acetanilid. Hydrogen dioxide is used in medicine as an antiseptic, and in the arts as an oxidizer. An illustration of the latter application is the custom, formerly in vogue, of bleaching dark hair a golden tint by the application of hydrogen dioxide. The disastrous effects of the continued treatment of this kind are now so well known that it is used less than it formerly was for this purpose.

Dose.—4 Cc. (1 fluidrachm).

THE HALOGENS

By the halogens we mean the four elements, chlorine, bromine, iodine, and fluorine. The word halogen is derived from two Greek words, *als*, which means a salt, and *eidos*, which means a likeness, the phrase being coined to illustrate the similarity of the compounds of these elements to salt, sodium chloride, which is taken as the type of haloid salts.

CHLORINE

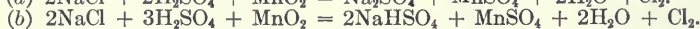
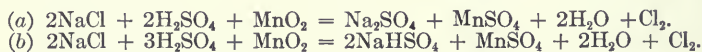
Symbol, Cl. Atomic weight, approximately 35.5

This element derives its name from the Greek word *chloros*, which means green, because of the greenish-yellow tint of the gas itself. Chlorine was isolated by Scheele in 1774, who obtained it from hydrochloric acid by treatment similar to that now in vogue in its manufacture. By reason of its origin Scheele called the new substance *dephlogisticated muriatic acid*, because he concluded it represented hydrochloric acid which had been freed from phlogiston, that hypothetical element which played so important a part in the chemical philosophy of the eighteenth century. Scheele considered the substance a compound, and it was not until 1810 that Sir Humphrey Davy proved it was an element, and by him it was named chlorine.

Chlorine is manufactured for pharmaceutic purposes by treatment of hydrochloric acid with magnesium dioxide, in which case the reaction is as follows:

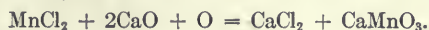


In practice, for the hydrochloric acid a mixture of common salt and sulphuric acid is usually substituted, the chemical action being by either of the following equations, according to relative proportion of chemicals used:



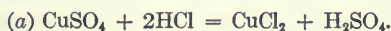
This process has the advantage over the first process in avoiding loss of hydrochloric acid, for it will be noticed that when hydrochloric acid is used, half of it goes to make manganese chloride, while in the second process the metals combine with the less expensive sulphuric acid.

An economic modification of the first process is found in *Weldon's regenerative process*, which consists in treating the manganese chloride with lime in the presence of air, the reaction being as follows:

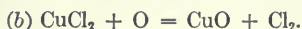


From the calcium chloride thus produced hydrochloric acid can be obtained, and from this the chlorine can be easily isolated, while from the calcium manganate, manganese dioxide is produced.

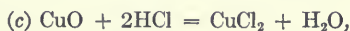
Chlorine is used so largely in the arts that the isolation of the element is one of the great industries especially in England. As in the case of such industries, persistent efforts are made to obtain more satisfactory and cheaper processes of manufacture. The Weldon regenerative process, above cited, was an illustration of the ingenuity used in attempting to cheapen the production of this staple; and investigators turned their efforts to the obtaining chlorine without the use of the comparatively expensive manganese salt. One chemical process of this character, which has the advantage of being practically continuous, is the *Deacon process*. In this process bricks or other porous pieces of earthenware are impregnated with a saturated solution of the sulphates of copper and sodium; after heating to redness in a furnace the vapors of hydrochloric acid are passed over them. The following reaction ensues:



The sulphuric acid of the above reaction is condensed, and is thus saved. The bricks, now impregnated with copper chloride, are then heated in a current of air, the reaction being as follows:



The chlorine vapors are then passed into the desired condensing agent, be it water or lime, while copper oxide remains in the brick. The bricks are then heated in a current of hydrochloric acid, the reaction being:



and the water is allowed to escape as steam. It will be seen that the bricks are impregnated once more with copper chloride, and from that time on they are treated with successive applications of air and hydrochloric acid vapor by the reactions *b* and *c*. Thus a comparatively small quantity of copper salt will be made to convert tons of hydrochloric acid into chlorine.

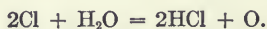
Of late chlorine has been produced by electrolytic methods, the Castner process consisting of passing electricity through a solution of sodium chloride, the negative pole being in a separate compartment, access to which is through a layer of mercury. In this way the salt is decomposed into chlorine and sodium hydroxide, whereas when the negative pole is in the same compartment as the positive, chlorinated soda is produced.

A method of obtaining chlorine at the prescription counter is by the treatment of potassium chlorate with hydrochloric acid. This process is less used for the isolation of chlorine than for the extem-

poraneous manufacture of a preparation similar to chlorine water, and it constitutes the official recipe for compound solution of chlorine U. S. P., and this has become a popular prescription in several sections of the country, the physician prescribing, instead of chlorine water, a solution of potassium chlorate containing hydrochloric acid. According to the method in which this prescription is compounded two very different results are obtained. If the potassium chlorate is dissolved in water and the hydrochloric acid added to the diluted solution, we obtain merely an acid solution of potassium chlorate. On the other hand, if the powdered potassium chlorate is placed in a bottle, the prescribed quantity of hydrochloric acid added, and the bottle allowed to stand corked a minute or two, the yellow fumes of chlorine are evolved, and on filling the bottle with water a variety of chlorine water is obtained. In receiving such a prescription the careful pharmacist is frequently in a quandary as to which method he shall pursue. In the writer's experience, several physicians objected to the chlorine water. Therefore the only advice that can be given is to call the attention of the prescriber to the two possibilities, and ask which result is desired. It is usual, however, that the method in which the chlorine is easily evolved is the desired method of compounding the prescription.

The Medical Use of Chlorine.—This represents but a small tithe of its commercial value, it being safe to say that tons of chlorine are used for bleaching and manufacturing purposes, where pounds of chlorine are used in the healing art. In medicine the use of chlorine is chiefly as a disinfectant.

Disinfectants are those bodies which destroy offensive organic matter—material which by decomposition breed disease. Most disinfectants are oxidizing agents, but chlorine is particularly interesting because it in itself is not a complete oxidizing agent. To be an oxidizing agent a body should contain oxygen which it can give up. Oxygen, of course, is not found in chlorine, but this energetic element performs the function by borrowing oxygen of its neighbor in this manner:



In other words, chlorine acts as an oxidizing agent only in the presence of water.

The above statement also explains the bleaching action of chlorine. The use of chlorine as a bleach was discovered by Berthollet in the year 1788, and the process proved so much more satisfactory for the finishing of textile fabrics than sun-bleaching, then in vogue, that its use soon became extended. At first the various cloth factories produced their own chlorine used in bleaching, but since the manufacture of chlorine was a matter so different from that of the manufacture of cloth, it soon fell into the hands of experts. The transportation of the chlorine to the cloth manufacturers soon became a serious matter. In those days the compression of gases had not reached the stage of perfection that now obtains, and at first the only practical method of dispensing was in the form of an aqueous solution, and the transportation of the large quantity of the water necessary to hold the small quantity of chlorine rendered this plan practically

prohibitive. The problem of a practical method of transportation of chlorine was found by Tennant, in the year 1799, in his discovery of chlorinated lime, it being found that freshly burnt lime was capable of absorbing as much as 35 to 40 per cent. of its weight of chlorine. From that time on the cloth manufacturers made use of this bleaching powder, and the manufacture of this chemical is now one of the most important industries in England. Chlorine is found in a loosely combined form in three preparations in the pharmacopœia—*compound solution of chlorine*, *chlorinated lime*, and *solution of chlorinated soda*.

LIQUOR CHLORI COMPOSITUS. Compound Solution of Chlorine.

[To replace AQUA CHLORI, PHARM. 1890]

An aqueous solution, containing, when freshly prepared, about 0.4 per cent. of Chlorine [$\text{Cl} = 35.18$], with some oxides of chlorine and potassium chloride.

<i>Recipe.</i> —Potassium Chlorate, granulated, five grammes....	5 Gm.
Hydrochloric Acid, eighteen cubic centimeters.....	18 Cc.
Distilled Water, a sufficient quantity,	
To make about one thousand cubic centimeters..	1000 Cc.

Add the Hydrochloric Acid, diluted with *twenty cubic centimeters* of Distilled Water, to the Potassium Chlorate contained in a flask of the capacity of about *two thousand cubic centimeters*. Insert in the flask a stopper perforated to admit a funnel of the capacity of about *one hundred cubic centimeters* containing about *ten grammes* of purified cotton well wetted with cold water; place the flask on a water-bath containing boiling water for a period of from two to three minutes; when the flask is completely filled with a greenish-yellow gas, remove it from the bath and add cold Distilled Water through the cotton in the funnel, in two separate portions of *five hundred cubic centimeters* each. After the addition of each separate portion of cold Distilled Water, stopper the flask securely, invert, and thoroughly agitate the contents.

This solution should be freshly made when wanted.

Remarks.—This new official replaces chlorine water of the pharmacopœia of 1890, and, like it, contains about 0.4 per cent. chlorine gas. While the formerly official chlorine water was made by passing chlorine gas into water, the present official is made by treating potassium chlorate with hydrochloric acid, and therefore contains oxides of chlorine and potassium chloride.

Dose.—4 Cc. (1 fluidrachm).

CALX CHLORINATA. Chlorinated Lime.

CHLORINATED CALCIUM OXIDE.

[CALX CHLORATA, PHARM. 1890]

A compound resulting from the action of chlorine upon calcium hydroxide, and containing not less than 30 per cent. of available chlorine. It is often improperly called "Chloride of Lime." It should be kept in well-closed vessels, in a cool and dry place.

A white, or grayish-white, granular powder, exhaling the odor of hypochlorous acid, having a repulsive, saline taste, and becoming moist and gradually decomposing on exposure to air.

In water or in alcohol it is only partially soluble.

Test for Identity.—Chlorine, given above. Calcium ammonium oxalate gives white precipitate. See Part V.

Assay.—See Part V.

Remarks.—Chlorinated lime is defined as a compound resulting

from the action of chlorine upon calcium hydroxide, and containing not less than 30 per cent. of available chlorine. The requirement of the pharmacopœia of 1890 (35 per cent. of available chlorine) was unduly high, experience showing that practically no chlorinated lime of strength greater than 30 per cent. can be found in the market; hence at the last revision the requirement was cut to 30 per cent. As mentioned above, chlorinated lime is made by the passage of chlorine gas over unslaked lime, the process being carried on, on a large scale, by passing the gas into specially constructed chambers in which the lime is arranged in different layers on special racks.

Chlorinated lime should not be called "chloride of lime," for it is not chloride of lime; that (or, more correctly, calcium chloride) having the formula CaCl_2 . Some authorities give the formula of chlorinated lime as CaOCl_2 , which gives the idea of a definite chemical compound, which the body is not. It is a loose indefinite mixture of calcium chloride, CaCl_2 , and calcium hypochlorite.

What is calcium hypochlorite? The calcium salt of hypochlorous acid, which comes from hypochlorous oxide.

And what is hypochlorous oxide? That oxide of chlorine in which the element shows its lowest valence.

And what is the valence of chlorine?

The valence of chlorine is I, III, V, and VII.

In hypochlorous oxide chlorine has the valence I, hence the formula of the oxide (oxygen having valence II) is Cl-O-Cl or Cl_2O . Hypochlorous acid is made by adding water to the oxide— $\text{Cl}_2\text{O} + \text{H}_2\text{O} = \text{H}_2\text{Cl}_2\text{O}_2 = 2\text{HClO}$. Calcium has the valence II—one atom replaces two hydrogen atoms of an acid. Since hypochlorous acid, HClO , has but one hydrogen atom and two are needed, we double the formula, making it, as above, $\text{H}_2\text{Cl}_2\text{O}_2$, and replacing the two hydrogen atoms by one calcium atom we get CaCl_2O_2 , or, as it is usually written, $\text{Ca}(\text{OCl}_2)$. This is the formula of calcium hypochlorite.

Chlorinated lime is a mixture of calcium chloride (CaCl_2) and calcium hypochlorite (CaO_2Cl_2). Let us add these—

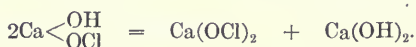
Calcium chloride..... Ca Cl_2

Calcium hypochlorite..... $\text{Ca O}_2\text{Cl}_2$

The sum is..... $\text{Ca}_2\text{O}_2\text{Cl}_4$, or 2CaOCl_2 , for which the graphic formula $\text{Ca} \begin{smallmatrix} \diagup \text{OCl} \\ \diagdown \text{Cl} \end{smallmatrix}$ has been assigned.

There are few chemicals regarding whose composition there has been more discussion and less resultant knowledge. Besides the two formulas just given, there have been a number of other suggestions as to its composition.

Thus, in order to explain why a large residue of calcium hydroxide is left when the chemical is extracted with water (for a mixture of calcium chloride and calcium hypochlorite would be completely soluble), it has been claimed that chlorinated lime is a mixture of calcium chloride with a body $\text{Ca} \begin{smallmatrix} \diagup \text{OH} \\ \diagdown \text{OCl} \end{smallmatrix}$. This body is supposed to dissociate when treated with water as follows:



Is it not possible that chlorinated lime is a solution or physical combination of chlorine gas in calcium hydroxide plus more or less calcium chloride?

This would explain how calcium hydroxide remains when water is added; this would also explain why chlorinated lime constantly loses chlorine on standing.

Dose.—250 milligrammes (4 grains).

LIQUOR SODÆ CHLORINATÆ. Solution of Chlorinated Soda.

[LIQUOR SODÆ CHLORATÆ, PHARM. 1890]

An aqueous solution of several chlorine compounds of sodium, containing at least 2.4 per cent., by weight, of available chlorine.

A clear, pale greenish liquid, having a faint odor of chlorine, and a disagreeable, alkaline taste.

Specific gravity: about 1.050 at 25° C. (77° F.).

The Solution at first colors red litmus-paper blue, and then bleaches it.

The addition of hydrochloric acid to the Solution causes an evolution of chlorine and carbon dioxide.

Recipe.—For full details of manufacture, see Part VII.

Assay.—See Part V.

Remarks.—Solution of chlorinated soda is directed by the pharmacopœia to contain 2.4 per cent. of chlorine. The recipe of the pharmacopœia of 1890 was an impossible one, that standard demanding the manufacture of the solution from chlorinated lime, the three stages of the process involving two filtrations and one warming, with a loss of but $\frac{2\frac{5}{8}}{2\frac{5}{8}}$ of the original chlorine.

The process of the present pharmacopœia is an improvement, inasmuch as the chlorine strength has been reduced from 2.6 per cent. available chlorine to 2.4 per cent.

Solution of chlorinated soda is called *Labarraque's solution*, after the French chemist who devised the original formula. *Javelle water*, or solution of chlorinated potassa, was the original preparation, but experience showed Labarraque's solution to be the more satisfactory preparation, hence Javelle water has fallen into disuse. Note that these two preparations are called solutions of *chlorinated* soda and potassa, with the express intention of emphasizing the uncertainty of the chemical character, and it may be here stated that these two preparations, as well as the chlorinated lime above mentioned, are now considered as irregular solutions of chlorine in the alkalis rather than as definite chemicals.

Dose.—1 Cc. (15 minims).

BROMUM. Bromine.

Br = 79.36

It should contain not less than 97 per cent. of pure Bromine, and be kept in protected, glass-stoppered bottles, in a cool place.

A heavy, dark brownish-red, mobile liquid, evolving, even at ordinary temperatures, reddish fumes, highly irritating to the eyes and lungs, and having a peculiar suffocating odor, resembling that of chlorine.

Specific gravity: 2.990 to 3.000 at 15° C. (59° F.). Boiling-point, 63° C. (145.4° F.).

Soluble in 28 parts of water at 25° C. (77° F.), and readily soluble in alcohol or ether (with gradual decomposition of these liquids); also in carbon disulphide and in chloroform, with a deep reddish-yellow color.

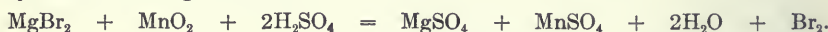
On exposure to air or heat it is completely volatilized without leaving any residue.

It destroys the color of solutions of litmus and indigo, and imparts a yellow color to solution of starch.

If Bromine be added to an excess of potassium hydroxide T.S., it should combine to form a permanently clear liquid, without the separation of oily drops (absence of *organic bromine compounds*).

If an aqueous solution of Bromine be shaken with a slight excess of reduced iron until it becomes nearly colorless, the filtered liquid, on the addition of a small amount of ferric chloride and of starch T.S., should not assume a blue color (absence of *iodine*).

Remarks.—Bromine was isolated by Balard in 1826 from water of the Mediterranean. On account of the very irritating odor it possesses, the discoverer named it after the Greek word *bromos*, which means a stench. Bromine is made from the bromides by a process analogous to that for the isolation of chlorine—*i. e.*, by treatment of the bromide with sulphuric acid and manganese dioxide—a sufficient quantity of acid is added so that at the end of the reaction the manganese will be of the form of a sulphate, thus insuring complete isolation of the bromine. As the source of bromine a native brine called *bittern* is generally used. This *bittern* is found in various sections of the world, one of the chief sources of supply being salt wells, found in the southwest of West Virginia and in the adjacent territory of southeast Ohio and western Pennsylvania. This *bittern* contains sulphates, bromides, and chlorides of the alkalies, chiefly of magnesium, and a crude concentration of the brine causes the crystallization of the less soluble sulphates and chlorides, leaving a mother liquid consisting chiefly of magnesium bromide. From this mother liquid bromine is obtained by the following reaction:



In addition to the explanation of the reaction given above it need only be added that the bromine, on distilling from the reacting mixture, is condensed in earthenware worms, and enters the market after purification by redistillation. Bromine is a very heavy brown liquid, specific gravity 3.000, having a disagreeable and irritating odor. The corrosive action of its vapor renders it necessary to use care in handling, as it is apt to cause injury to the mucous membrane of the nose and throat and also to the delicate tissue of the eye. The liquid, bromine, is decidedly caustic, hence the operator must be careful to avoid dropping same on his hands or face. Bromine is used in medicine in a free state for practically but one preparation, the *Bibron antidote for rattle-snake poison*, and its use in this way is rather limited.

It is, of course, largely used in the preparation of the very popular bromides. On treating a solution of soda with bromine we obtain solution of sodium hypobromite, which is analogous to the Labarraque's solution just described, and is used instead of Labarraque's solution in determination of urea.

IODUM. Iodine.

I = 125.90

It should contain not less than 99 per cent. of pure Iodine, and be kept in glass-stoppered bottles, in a cool place.

Heavy, bluish-black, dry and friable, rhombic plates, having a metallic lustre, a distinctive odor, and a sharp acrid taste.

Specific gravity: 4.948 at 17° C. (62.6° F.).

Iodine imparts a deep brown, evanescent stain to the skin, and slowly destroys vegetable colors.

Soluble in about 5000 parts of water, and in 10 parts of alcohol at 25° C. (77° F.); freely soluble in ether, chloroform, or carbon disulphide; its solution in alcohol or in an aqueous solution of potassium iodide has a reddish color; its solution in chloroform or carbon disulphide has a violet color.

It volatilizes slowly at ordinary temperatures. When heated to about 114° C. (237.2° F.) it fuses, and is gradually dissipated in the form of a purple vapor, leaving no residue.

With starch T.S. a dark blue color is produced.

A solution of iodine in chloroform should be perfectly clear and limpid (absence of *moisture*).

To determine the presence of cyanogen, chlorine, or bromine, proceed as follows:

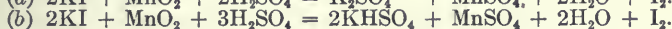
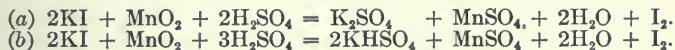
Triturate 0.5 Gm. of finely powdered Iodine with 20 Cc. of water, and filter the solution. To one-half of this solution, in a test-tube, carefully add tenth-normal sodium thiosulphate V.S., until the solution is just decolorized. Then add a few drops of ferrous sulphate T.S., and subsequently a little sodium hydroxide T.S., and heat the mixture gently. On now adding a slight excess of hydrochloric acid, the liquid should not assume a blue color (absence of *iodine cyanide*).

To the other half of the aqueous filtrate, in a test-tube, add a slight excess of silver nitrate T.S., shake the liquid actively, allow the precipitate to subside, and, having poured off the clear supernatant liquid completely, shake the precipitate with a mixture of 1 Cc. of ammonia water and 9 Cc. of water, and filter. Upon the addition of a slight excess of nitric acid to the filtrate, not more than a slight opalescence should make its appearance (limit of *chlorine* or *bromine*).

Test for Impurities.—Impurities just given above.

Assay.—Given in Part V.

Remarks.—This element was discovered by Courtois in the year 1811, and the discovery was announced by Clement before the National institute November 29, 1813. It was named by Gay Lussac (1813) after the Greek word *iodos*, meaning violet, on account of the violet tint of its vapor. Iodine was formerly obtained entirely from the ashes of marine plants. In some sections of the world, notably the North of Ireland and on the Scotch coast, these weeds were collected in considerable quantities, and after being dried, were burned, the ashes being called "*kelp*" in Ireland, and "*varec*" in Normandy. These ashes contain iodides, and by treatment with manganese dioxide and sulphuric acid they yield iodine by either of the following reactions:



In both these cases the iodine is separated by sublimation.

In other sections other methods of isolation are employed, such as treatment of the ashes with chlorine, nitrous acid, ferrous and copper sulphates. Moreover, processes are now in vogue in which an infusion of the sea-weed is used instead of the ashes, these later and more economic processes being devised in order that the European product might compete with the great modern source of iodine, the nitre beds of Peru.

The Peruvian nitre consists chiefly of sodium nitrate (the so-called Chile saltpeter). This is obtained from the ore by lixiviation, and the saline solution is concentrated to the point at which the sodium nitrate will crystallize.

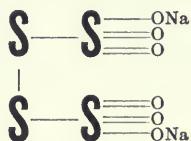
The mother liquor from this crystallization contains sodium iodate, which is obtained by evaporating the liquid to dryness. Iodine is isolated from this impure iodate, NaIO_3 , by treatment with chlorine (after conversion of the iodate into iodide by use of sulphur dioxide), or with nitrous acid or with a mixture of copper and ferrous sulphates and liberation of iodine from the cuprous iodide thus produced by treatment with manganese dioxide and sulphuric acid.

In all these South American processes except the last the crude iodine is separated from the other products by filtration. It is then purified by sublimation. Iodine is a blue-black, metallic-appearing solid, insoluble in water, but soluble in a solution of potassium iodide, in alcohol, in ether and chloroform, the solution in the last two solvents being a pink to purple tint. It is quite volatile, hence it must be preserved in well-closed bottles, and by reason of its volatility it is usually purified by sublimation, hence the trade name of the best quality of iodine is *resublimed iodine*.

The test for iodine is the fact that it turns mucilage of starch an intense blue-black color. When this test is applied for the detection of iodides it is necessary to liberate the iodine by treatment with some substance containing free chlorine. The iodides form a characteristic yellow precipitate of silver iodide when treated with a solution of silver nitrate, and this precipitate, unlike similar precipitates from chlorides and bromides, does not dissolve in ammonia water. This is the basis of the official test for distinction of bromides and chlorides given above. The aqueous and alcoholic solutions of free iodine have a brown tint, and such solutions are decolorized by treatment with sodium thiosulphate (hyposulphite, see p. 477), the equation being:



Both products of the reaction just given—sodium iodide and sodium tetrathionate—are colorless, and the latter chemical has the graphic formula—



The word "tetrathionate" means literally "four-sulph-ate," *tetra* being Greek for "four," and *thion* means "sulphur" in the same language.

This decoloration of iodine by sodium hyposulphite is applied in making the so-called *decolorized tincture of iodine*. As will be seen from above equation, the name is a misnomer, as the decolorized preparation contains no uncombined iodine, and for all purposes a solution of potassium or of sodium iodide would have equal therapeutic value.

The property of sodium hyposulphite of decolorizing iodine is applied for the quantitative estimation of that element and its derivatives. For this purpose a volumetric solution of sodium hyposulphite is dropped into the iodine solution until the latter is decolorized.

In order sharply to distinguish the end reaction, a starch paste

is used as an indicator, the blue-black tint in the mixture being discharged the moment a sufficient quantity of the volumetric solution is added. (See Chapter LIV.)

The official preparations of iodine are: *tincture of iodine*, an alcoholic solution containing 7 per cent. iodine and 5 per cent. potassium iodide (p. 228); *compound solution of iodine*, which contains 5 per cent. of iodine dissolved in water by aid of 10 per cent. of potassium iodide (p. 185); and the *ointment of iodine*, which contains 4 per cent. of iodine and 4 per cent. potassium iodide, rubbed with glycerin and benzoinated lard (p. 347).

Dose.—5 milligrammes ($\frac{1}{10}$ grain).

SULPHUR.

Symbol, S. Atomic weight, approximately 32

This element has been known from ancient times, the name being of Latin origin, derived from two words, *sal* (salt) and *pyr* (fire). Sulphur is found in the free state in the neighborhood of volcanoes, it being emitted through these from the interior of the earth. It is also found very widely distributed in combination, for example, *gypsum*, which is calcium sulphate, *iron pyrites* (ferrous sulphide), and *galena* (lead and silver sulphide). The commercial source of sulphur is chiefly crude brimstone, which is obtained either from the sides of volcanoes or else mined in certain sections of the world, as, for example, a very extensive deposit of sulphur in Louisiana.

Sulphur is extracted from the Louisiana mines by sinking several 4-inch steel tubes into the deposit, and passing superheated steam through one of the tubes. The heat of the steam melts the sulphur, while the steam pressure forces the fused sulphur through the other tubes into vats or ponds, where it solidifies.

The crude volcanic sulphur is roughly purified by straining and pouring into iron molds. Such sulphur comes into the market in cylindric pieces, one or two feet long and two or three inches in diameter, under the name of *roll sulphur* or *brimstone*. A purer commercial form is *sublimed sulphur* or *flowers of sulphur*, which is made by putting brimstone in cast-iron retorts and collecting the vapors in large, well-cooled chambers, on the floors of which sulphur falls in the form of fine powder.

Sulphur is a lemon-yellow body, and according to whether it is rolled sulphur or sublimed sulphur, is in lump or powder form. These represent one of several forms which the element assumes. Thus, if brimstone is fused and let stand until a pellicle forms on the surface of the fused mass, and then the liquid portion is poured off, the element is found in the form of *yellow prismatic crystals*.

A third form which the element assumes is prepared by taking either the crystals or the rolled sulphur, and heating it in a crucible to a temperature of 330° F., when it is poured into cold water, forming an elastic, rubber-like mass called *amorphous sulphur*. These three widely different bodies are sulphur and nothing else, and it might be said that the element assumes three different masquerades. Several elements are known to possess this same characteristic of assuming different forms, and such a phenomenon is called *allotropism*.

The official forms of sulphur are:

SULPHUR SUBLIMATUM. Sublimed Sulphur.

S = 31.83

It should contain not less than 99 per cent. of pure Sulphur.

A fine, yellow powder, having a slight characteristic odor, and a faintly acid taste.

Insoluble in water; readily soluble in carbon disulphide; slightly soluble in absolute alcohol; more readily soluble in petroleum benzin, benzene, oil of turpentine, and many other oils; also in ether, chloroform, and in boiling aqueous solutions of alkali hydroxides.

At about 115° C. (239° F.) it fuses to a yellow, mobile fluid, which upon further heating becomes dark and viscid, until a temperature above 300° C. (572° F.) is reached, when it becomes a thin liquid, boiling at 448° C. (838.4° F.). In the air it burns to sulphur dioxide, characterized by its odor, and by its blackening a strip of paper moistened with mercurous nitrate T.S. held in the gas.

When agitated with water, the latter gives an acid reaction with litmus-paper.

The amount of residue left after volatilizing or burning a weighed portion of well-dried Sublimed Sulphur should not exceed 0.5 per cent.

Dose.—4 Gm. (60 grains).

SULPHUR LOTUM. Washed Sulphur.

S = 31.83

Washed sulphur, when dried, should contain not less than 99.5 per cent. of pure Sulphur.

Recipe.—Sublimed Sulphur, one hundred grammes..... 100 Gm.
Ammonia Water, ten cubic centimeters..... 10 Cc.
Water, a sufficient quantity.

Pass the Sublimed Sulphur through a No. 30 sieve, mix it thoroughly with one hundred cubic centimeters of Water, add the Ammonia Water, and digest for three days, agitating occasionally. Then add one hundred cubic centimeters of Water, transfer the mixture to a muslin strainer, and wash the Sulphur with Water until the washings cease to impart a blue color to red litmus-paper. Then allow it to drain, press the residue strongly, dry it rapidly at a moderate heat, pass it through a No. 30 sieve, and keep it in well-stoppered bottles.

Tests for Identity.—A fine yellow powder without odor or taste. Rest of description identical with that for sublimed sulphur.

Residue on ignition not more than $\frac{2}{10}$ of 1 per cent.

Test for Impurities.—Earthy and metallic impurities:

If 0.5 Gm. of Washed Sulphur be boiled with 10 Cc. of sodium hydroxide T.S., it should be dissolved, leaving no appreciable residue (absence of earthy and metallic impurities).

Arsenic: Gutzeit's test, see Part V.

Acid or ammonia:

If 5 Cc. of water be agitated with 2 Gm. of Washed Sulphur, the liquid should not change the color of blue or red litmus-paper (absence of acid and of ammonia).

SULPHUR PRÆCIPITATUM. Precipitated Sulphur.

S = 31.83

Precipitated Sulphur, when dried, should contain not less than 99.5 per cent. of pure Sulphur.

Recipe.—Sublimed Sulphur, one hundred grammes..... 100 Gm.
Lime, fifty grammes..... 50 Gm.
Hydrochloric Acid,
Water, each, a sufficient quantity.

Slake the Lime, and mix it uniformly with *five hundred cubic centimeters* of Water. Add the Sublimed Sulphur, previously sifted, and, after thorough mixing, add *one thousand cubic centimeters* of Water, and boil the mixture in a porcelain or glass vessel during one hour, stirring or agitating very frequently, and replacing the Water lost by evaporation. Then cover the vessel, and permit the contents to cool and become clear by subsidence. Carefully draw off the clear solution, and filter the remainder. To the united liquids add gradually, and with constant stirring, Hydrochloric Acid, previously diluted with an equal volume of Water, until the liquid is nearly neutralized, still retaining, however, an alkaline reaction and a yellow color. Collect the precipitate on a strainer, and wash it, until the washings are tasteless and cease to give a precipitate upon the addition of ammonium oxalate T.S. Then dry the product rapidly, at a moderate heat, and keep it in well-stoppered bottles.

A fine, amorphous powder, of a pale yellow color, without odor or taste.

Insoluble in water; very slightly soluble in absolute alcohol; readily soluble in carbon disulphide, petroleum benzin, benzene, oil of turpentine, and many other oils; also in ether, chloroform, and in boiling aqueous solutions of alkali hydroxides.

At 115° C. (239° F.) Precipitated Sulphur melts, and at a higher temperature it volatilizes, or, if air be admitted, burns to sulphur dioxide.

If 1 Gm. of Precipitated Sulphur be ignited, it should not leave a weighable residue.

Impurities.—*Arsenic:* Gutzeit's test, see Part V.

Acid or ammonia: As in washed sulphur.

Remarks.—Washed sulphur is the sublimed sulphur from which the sulphurous and sulphuric acid have been removed by washing with a diluted solution of ammonia.

In subliming sulphur there is always sufficient air present to effect a slight oxidation of the sulphur, which means the production of sulphurous oxide, SO_2 , and sulphuric oxide, SO_3 , and these oxides pass over with the vaporized sulphur and cling to it on solidification. Hence almost all sublimed sulphur contains traces of acid impurities. These impurities produce griping effect when contaminated sulphur is administered, and, accordingly, the removal of these traces of acid is advisable. This is accomplished by washing the sulphur with volatile alkali,—ammonia water,—this possessing the great advantage over the fixed alkalies (sodium hydroxide or potassium hydroxide) of being easily separated from the sulphur, simple exposure to air removing the last traces.

Precipitated sulphur is made by precipitating sulphur from its alkaline solution by the addition of hydrochloric acid, the equation of the two stages of the reaction being:



The equations can be explained as follows: We prepare a solution of sulphur by chemically combining it with calcium oxide, whereby there is produced a soluble mixture of the polysulphides of sulphur, CaS_2 to CaS_5 , and calcium thiosulphate, CaS_2O_3 . This sulphur solution is filtered, and to the clear liquid is added another clear liquid, hydrochloric acid, when a copious precipitate occurs, the acid seizing the calcium to form calcium chloride, and thus goes into solution, while the sulphur is precipitated. If, in the above reaction, sulphuric acid is used as a precipitant instead of hydrochloric acid, we obtain a

precipitate consisting of sulphur contaminated with calcium sulphate. This body is known in commerce as *lac sulphur*, or milk of sulphur. The pharmacist must bear in mind that this does not represent the pharmacopœial precipitated sulphur, which one should specify by its official name in ordering. A simple test whereby to distinguish the official washed sulphur from the milk of sulphur is that the former, when burned on the end of a spatula, should yield no residue, whereas after burning the milk of sulphur there will remain a considerable quantity of calcium sulphate.

Dose.—4 Gm. (60 grains).

SULPHURIS IODIDUM. Sulphur Iodide.

Recipe.—Washed Sulphur, twenty grammes..... 20 Gm.
Iodine, eighty grammes..... 80 Gm.

Mix the Sulphur and Iodine thoroughly by trituration; introduce the mixture into a flask, close the orifice loosely, and, by means of a water-bath, gradually and with occasional agitation apply a heat not exceeding 60° C. (140° F.), until the ingredients combine and become of a uniformly dark color throughout. Then increase the heat to the boiling-point of water, so as to fuse the mass. Should any Iodine have sublimed and condensed on the glass, incline the flask so as to combine the Iodine with the fused mass, and then pour the latter upon a porcelain plate or other suitable cold surface. When it is cold, break the product into pieces of suitable size, and keep them in a glass-stoppered bottle, in a cool place.

Brittle masses of a crystalline fracture and a grayish-black, metallic luster, having the odor of iodine and a somewhat acrid taste.

Almost insoluble in water; soluble in about 60 parts of glycerin; very soluble in carbon disulphide. Alcohol, ether, and a solution of potassium iodide dissolve the iodine, leaving the sulphur. Continued boiling with water vaporizes all of the iodine, leaving about 20 per cent. of sulphur as residue.

On exposing Sulphur Iodide to the Air, it gradually loses iodine. On heating it, some iodine sublimes at first; at a somewhat higher temperature a sublimate is formed, containing both iodine and sulphur. At a still higher temperature, the whole is volatilized, leaving only a trace of residue.

Assay.—See Part V.

Remarks.—Iodized sulphur is an indefinite mixture, since it loses iodine on standing. It has been claimed to possess the formula S_2I_2 , which can be explained by the following structural formula:



In this it will be noticed that we have two atoms of bivalent sulphur; one bond of each is used in holding together its neighboring sulphur atom. Thus, each atom of sulphur has only one bond to which iodine is linked. No chemical formula is assigned sulphur iodide in the pharmacopœia. This is because there are grave doubts as to its being a definite chemical compound, and it may be wiser to call it "iodized sulphur" or "iodated sulphur," just as the pharmacopœia has adopted the terms chlorinated lime and sulphurated lime in place of the old name of these bodies, chloride of lime and sulphide of calcium.

The method of the manufacture of this body given by the pharmacopœia of 1890 was by melting the iodine and sulphur together in a

test-tube or flask, an impracticable process, inasmuch as a large amount of the iodine will have evaporated before the mixture is brought to a state of fusion sufficient to pour it out. The present recipe seeks to lessen loss of iodine by heating a mixture of sulphur and iodine on the water-bath before attempting fusion. A far better process is that of Prunier, who first brings the sulphur to a state of fusion and then sprinkles the iodine in the fused mass.

Sulphur iodide is used as an alternative in doses of 1 to 3 grains.

CARBONEI DISULPHIDUM. Carbon Disulphide.

$$\text{CS}_2 = 75.57$$

Carbon Disulphide should be kept in partially filled, well-stoppered bottles, or in tin cans, in a cool place, remote from lights or fire.

A clear, colorless, highly refractive liquid, very diffuse, having a strong, characteristic, but not fetid odor, and a sharp, aromatic taste.

Specific gravity: 1.256 to 1.257 at 25° C. (77° F.).

Soluble in 526 parts of water at 25° C. (77° F.); very soluble in alcohol, ether, chloroform, fixed and volatile oils.

Carbon disulphide vaporizes rapidly at the ordinary temperature, is highly inflammable, boils at 46° to 47° C. (114.8° to 116.6° F.), and when ignited, burns with a bluish-white flame, producing carbon dioxide and sulphur dioxide.

It should not affect the color of blue litmus-paper moistened with water (absence of *sulphur dioxide*).

A portion evaporated spontaneously in a glass vessel should leave no residue (absence of *dissolved sulphur*). Lead acetate T.S. agitated with it should not be blackened (absence of *hydrogen sulphide*).

Remarks.—This body is made by passing the vapors of sulphur through carbon contained in a red-hot tube or cylinder.

Carbon disulphide is a heavy, mobile, inflammable liquid, with the offensive odor of rotten eggs. It is said to be odorless when absolutely pure, a happy condition never as yet attained by the writer, although he has often rectified the disulphide for analytic work.

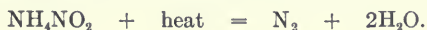
This rectification can be performed by the ordinary process of distillation, with the one safeguard of preventing direct contact of the distilling flask with flame. For this reason it is even unadvisable to use a flame for heating the water on which the distilling flask rests. If a steam-bath is not at hand, the distillation must be carried on by pouring heated water into the bath, rather than by directly heating the water. As carbon disulphide boils at 47° C., the occasional filling of the water-baths with boiling water will very rapidly volatilize all the carbon disulphide. The chief use of carbon disulphide is as a solvent for India-rubber, and it is for this reason that it is official, being used to dissolve rubber in the manufacture of the official mustard-paper.

NITROGEN

Symbol, N. Atomic weight, approximately 14

Nitrogen was discovered as a constituent of the air by Rutherford in 1772. The name nitrogen was coined by Chaptal in 1796 from the two words *nitron*, nitre, and *gennao*, I produce; investigation having shown that it was a constituent of nitre or saltpeter.

Nitrogen is usually prepared on the small scale by heating ammonium nitrite, the equation of reaction being:

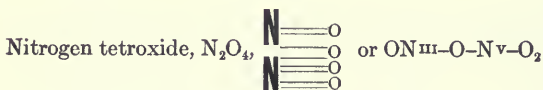
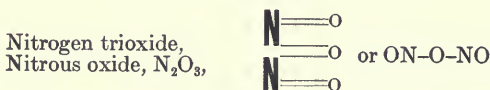
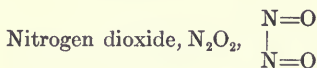
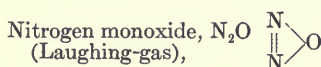


It is now being prepared from the atmosphere by electrolytic processes.

Nitrogen is a colorless, tasteless, odorless, and inert gas, which is of no pharmaceutical value. Its chief occurrence in uncombined state is in the atmosphere, of which it constitutes about 77 per cent. by weight and 79 per cent. by volume, and in which it does the inestimable service of diluting the very active constituent, oxygen. It has been proved that air is a mechanical mixture of nitrogen and oxygen, along with a small quantity of carbonic oxide, moisture, and traces of salts.

Nitrogen forms five chemical combinations with oxygen, and of these five oxides, only two follow the simple rules of valence.

The graphic formulas of the five are supposed to be as follows:

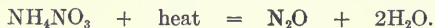


Of these five oxides, two—nitrous oxide, N_2O_3 , and nitric oxide, N_2O_5 —will be considered in the next chapter among the acids. Of the other three, the following comment will suffice:

Nitrogen monoxide, N_2O , is called *laughing-gas*, and also, unfortunately, *nitrous oxide*.

The latter synonym should fall into disuse, as that term more properly belongs to nitrogen trioxide, N_2O_3 , as that is the oxide from which nitrous acid is derived.

Nitrogen monoxide is made by heating ammonium nitrate, as shown in the following equation:



It is a colorless gas, of faint odor and sweetish taste, and is administered by inhalation by dentists as a mild anesthetic.

Nitrogen dioxide, N_2O_2 , is incorrectly called nitric oxide, as that name more properly belongs to nitrogen pentoxide, N_2O_5 , since that is the oxide from which nitric acid is derived.

Nitrogen dioxide is the colorless gas produced in a nitrometer when ethyl nitrite or other nitrites are treated with potassium iodide and sulphuric acid. (See assay of spirit of nitrous ether, p. 662.) It can be studied only in an inclosed apparatus like a nitrometer, for as soon as it comes in contact with the atmosphere it is converted into nitrogen tetroxide.

For this reason, while it is produced in oxidations with nitric acid (see Manufacture of Solution of Ferric Sulphate, p. 562), it can never be detected when such reactions occur in the air, where, instead of the colorless nitrogen dioxide, the red-brown fumes of nitrogen tetroxide are evolved.

Nitrogen tetroxide, N_2O_4 , is of interest to us only because a product of oxidation with nitric acid just mentioned.

It is a heavy, red-brown, irritating vapor, and can be easily produced by pouring nitric acid on copper turnings.

It is claimed by some writers that N_2O_4 is colorless, and that the red-brown gas has the formula NO_2 .

PHOSPHORUS. Phosphorus.

P = 30.77

It should contain not less than 99.5 per cent. of pure Phosphorus, and be carefully kept under water, in strong, well-closed vessels, in a secure and moderately cool place, protected from light.

A translucent, nearly colorless solid, of a waxy luster, having, at ordinary temperatures, about the consistence of beeswax. By long keeping the surface becomes white or red, and occasionally black. It has a distinctive and disagreeable odor and taste (*but should not be tasted, except in very dilute solution*). When exposed to the air, it emits white fumes, which are luminous in the dark, and have an odor somewhat resembling that of garlic; on longer exposure to air it often takes fire spontaneously.

Specific gravity: 1.830 at $10^\circ C.$ ($50^\circ F.$), and 1.820 at $25^\circ C.$ ($77^\circ F.$).

Melting-point: $44^\circ C.$ ($111.2^\circ F.$).

Phosphorus is insoluble, or nearly so, in water, to which, however, it imparts its characteristic disagreeable odor and taste; soluble in 350 parts of absolute alcohol at $15^\circ C.$ ($59^\circ F.$), in 240 parts of boiling absolute alcohol, in 80 parts of absolute ether, in about 50 parts of any fatty oil, and in about 25 parts of chloroform; it is very soluble in carbon disulphide, the latter yielding a solution which must be handled with the greatest care to prevent evaporation, which would be followed by instant ignition.

To test for *arsenic* and *sulphur* proceed as follows: Add 1 Gm. of Phosphorus to 10 Cc. of nitric acid diluted with 10 Cc. of distilled water, in a flask having a capacity of 50 Cc., and digest the mixture at a gentle heat on a water-bath while passing a current of carbonic acid gas into the flask over the surface of the liquid until the Phosphorus is dissolved. Transfer the solution to a dish, and evaporate it until no more nitrous vapors are given off, and then dilute the solution to 100 Cc. with distilled water. One Cc. of this solution should not respond to the Modified Gutzzeit's Test for *arsenic*, see p. 973.

On adding barium chloride T.S. to the remainder of the liquid, not more than a slight opalescence should be produced (limit of *sulphur*).

Remarks.—The name of this element is derived from the two Greek words, *phos*, light, and *pherein*, to bring, hence the name light-carrier, designating the prominent characteristic of phosphorus, the term having been used before the discovery of phosphorus for other phosphorescent bodies.

Phosphorus was discovered by Brandt in 1669. This alchemist, in examining urine, placed some in a retort and subjected it to distillation. The residue in the retort, after the distillation, became charred, and the charcoal acted on the phosphates present, isolating free phosphorus, which appeared in the retort with its characteristic phosphorescence.

The manufacture of phosphorus from urine was in vogue for one hundred years, it being first prepared from bones by Scheele, in 1771, the element being discovered in bones by Gahn a few years earlier.

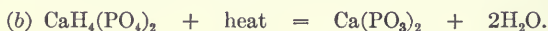
Phosphorus is never found in the free state in nature, as contact with the air causes a combination of it with the atmospheric oxygen.

It is found abundantly in the form of calcium phosphate in many minerals, such as *apatite*—calcium chlorophosphate and the “phosphate rock” of the southern Atlantic States. Calcium phosphate is also the main inorganic constituent of the bones, bone-ash containing 86 per cent. of that chemical.

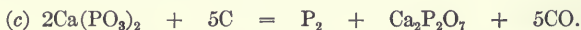
Phosphorus is made from bone-ash, there being several stages in the process. The bone-ash is first treated with sulphuric acid, thus forming calcium sulphate and acid calcium phosphate, as shown in the following equation:



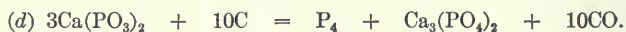
In passing, it might be mentioned that the acid calcium phosphate just cited is the constituent of the popular “*acid phosphates*,” and such preparations are made by this method, as will be seen by referring to the recipe for *liquor acidi phosphorici* of the National Formulary. In making phosphorus the acid phosphate obtained in the above reaction is then submitted to a high heat, whereby it loses water and is converted into calcium metaphosphate, as shown in the following equation:



This calcium metaphosphate is then heated with charcoal in a retort, and is decomposed into phosphorus and calcium pyrophosphate, as shown in the following equation:



A better yield of phosphorus can be obtained by using the larger proportion of carbon suggested in the following equation, thus getting calcium phosphate instead of calcium pyrophosphate:



In practice the reactions *b* and *c* are conducted together, and the isolated phosphorus is separated from the calcium pyrophosphate (or phosphate) by distillation, the distilled phosphorus collected under warm water and run into molds while still fused.

Phosphorus is now being manufactured by heating calcium phosphate with charcoal and sand in the electric furnace, under an atmosphere of coal-gas (Parker process).

Phosphorus is interesting as assuming the characteristic allotropic forms—the yellow, the red, and the metallic. *Phosphorus* (U. S. P.) is the yellow variety, and usually comes in commerce in cylindric sticks which must be kept under water or otherwise protected from the action of the atmosphere. This yellow phosphorus is as soft as wax, melts at 44° C., is soluble in absolute alcohol, ether, chloroform, and fixed oils. Exposed to the air, yellow phosphorus catches fire spontaneously, emitting dense white fumes of phosphoric oxide, P_2O_5 . This property renders it necessary to handle phosphorus with the utmost care, and in weighing same for pharmaceutic purposes it should always be done in water; *i. e.*, the stick of phosphorus should be cut under water contained in an evaporating dish, and weighed, not directly on the scale-pan, but in a beaker glass containing water.

That the weight of the beaker glass and the water should be previously determined, goes without saying.

As mentioned above, the yellow phosphorus, even under water, emits a faint, whitish light, which is supposed to be due to an evolution of minute quantities of phosphine, PH_3 , and the immediate decomposition of same with the evolution of a microscopic flame.

The red phosphorus is prepared by heating the yellow variety to 300°C . in closed tubes, in an atmosphere of nitrogen. This product is a dark-red powder, perfectly stable in air, and comparatively non-toxic. Red phosphorus is largely used in place of yellow phosphorus in the manufacture of matches and the like, and its use has greatly lessened the dangers of this trade.

Metallic phosphorus is made by heating the red to 530°C . in vacuo. It appears in steel-like scales, and has no interest in pharmacy.

Medical Properties.—Powerful nervous stimulant; a violent poison.

Dose.—0.5 milligramme ($\frac{1}{128}$ grain).

Antidote.—Old (ozonized) oil of turpentine.

Official Preparation.—Pills of phosphorus, each containing about $\frac{1}{160}$ grain of phosphorus (p. 324).

The pharmacopœia of 1890 also recognized:

Phosphorated oil (U. S. P. 1890)—1 minim represented about $\frac{1}{115}$ grain of phosphorus.

Spirit of phosphorus (U. S. P. 1890) solution of phosphorus (1.2 Gm.) in absolute alcohol (1000 Cc.).

Elixir of phosphorus (U. S. P.), made from the spirit and containing about $\frac{1}{65}$ grain of phosphorus to the fluidrachm.

CARBON

Symbol, C. Atomic weight, approximately 12

The name of this element is derived from the word *carbo*, which was the Latin name for coal.

Carbon is found in the free form as diamond, graphite, and coal, while its combinations are very widely distributed. For example, all organic matter contains carbon. It is also found as carbonate in several well-known minerals; thus, marble and lime-stone are calcium carbonate, while dolomite is a mixture of calcium and magnesium carbonates.

Carbon, as such, is found in three allotropic forms, they being diamond, graphite, and coal. *Diamond* is the crystalline form of carbon, and is found in hard, transparent masses, possessing the specific gravity 3.5 to 3.6. The proof of its being carbon pure and simple is that when ignited in a stream of oxygen it is all consumed with the exception of a trace of ash (about $\frac{1}{10}$ of 1 per cent.), forming nothing but carbon dioxide gas. Its formation from the other varieties of carbon has already been explained on p. 146.

Graphite is a gray-black, soft, amorphous solid, known to all as the so-called "lead" of the pencil. It has a specific gravity of about 2.25, and is of interest because of its resistance to heat. For this reason graphite is used for making crucibles—the so-called black-lead crucibles. (See p. 81.)

Coal is the impure, non-crystalline form of carbon, and can be divided into two commercial varieties, *anthracite*, or hard coal, *bituminous*, or soft coal. Coal, on being heated in retorts, gives up its volatile portion, yielding illuminating gas, ammonia, and coal-tar, while the residue is the commercial *coke*, which contains about 91 per cent. carbon. The great value of coal and coke as a fuel has already been touched upon on p. 68.

Charcoals are more or less pure forms of amorphous carbon.

CARBO LIGNI. Charcoal.

Charcoal prepared from soft wood, and very finely powdered. It should be kept in well-closed vessels.

A black, odorless, and tasteless powder, free from gritty matter.

If 1 Gm. of Charcoal be boiled with a mixture of 3 Cc. of potassium hydroxide T.S. and 5 Cc. of water for several minutes, the filtrate should be colorless or nearly so (evidence of *complete carbonization*).

Remarks.—This is to be prepared from soft wood, very finely powdered; that prepared from willow-wood being considered best.

The pharmacopœia directs that the wood-charcoal be kept in well-closed vessels, because it readily absorbs odors. In fact, the medical value of wood-charcoal depends very largely on this property of absorbing gases, and it is for this reason that charcoal is valuable in indigestion. The process of manufacture is given on p. 847.

Dose.—1 Gm. (15 grains).

CARBO ANIMALIS. Animal Charcoal.

Charcoal prepared from bone.

Dull black, granular fragments, or a dull black powder, odorless, nearly tasteless, and insoluble in water or alcohol.

When ignited, it leaves a grayish or yellowish-white ash, amounting to about 85 per cent. of the original weight of the portion taken, which should have been previously dried at 120° to 125° C. (248° to 257° F.) to a constant weight.

The ash should be soluble in hydrochloric acid with the aid of heat, leaving not more than a trifling residue.

If 1 Gm. of Animal Charcoal be boiled for several minutes with a mixture of 3 Cc. of potassium hydroxide T.S. and 5 Cc. of water, the filtrate should be colorless or nearly so (evidence of *complete carbonization*).

Remarks.—This form, commercially called *bone-black*, is prepared by the carbonization of bones, the dried bones being placed in a cast-iron retort and carbonized (during the process a liquid distils over which is called *bone oil*), and the residue in the retort is then removed and reduced to powder. This animal charcoal contains the carbon from the organic matter of the bones plus all the bone-ash, and it therefore contains a large amount of calcium phosphate. Animal charcoal is used as a decolorizer, as explained on p. 137, and the presence of calcium phosphate in the animal charcoal renders its use inadvisable in removing color from liquids containing acids, as calcium phosphate will dissolve therein. Therefore the pharmacopœia directs the use of the second form of animal charcoal, from which bone-ash has been removed.

CARBO ANIMALIS PURIFICATUS. Purified Animal Charcoal.

Recipe.—Animal Charcoal, in No. 60 powder, one hundred

<i>grammes</i>	100 Gm.
Hydrochloric Acid, three hundred grammes.....	300 Gm.
Boiling Water, a sufficient quantity.	

Introduce the Animal Charcoal into a capacious vessel, add *two hundred grammes* of Hydrochloric Acid and *four hundred cubic centimeters* of Boiling Water. By means of a sand-bath keep the mixture gently boiling during eight hours, adding water occasionally to maintain the original volume. Then add *five hundred cubic centimeters* of Boiling Water, transfer the mixture to a muslin strainer, and, when the liquid has run off, return the Charcoal to the vessel. Add to it *one hundred*

grammes of Hydrochloric Acid and two hundred cubic centimeters of Boiling Water, boil for two hours, again add five hundred cubic centimeters of Boiling Water, transfer the whole to a plain filter, and when the liquid has passed through, wash the residue with Boiling Water until the washings produce only a faint cloudiness with silver nitrate T.S. Dry the powder in a drying oven, and immediately transfer it to well-stoppered vials.

A dull black powder, odorless, tasteless, and insoluble in water, alcohol, or other solvents.

If 2 Gm. of the powder be ignited at a red heat with free access of air in a broad, shallow, porcelain or platinum dish it should not leave a residue weighing more than 0.08 Gm., or 4 per cent., of the original weight (limit of *silicates* and *other fixed inorganic matter*).

If 1 Gm. of the powder be boiled with a mixture of 3 Cc. of potassium hydroxide T.S. and 5 Cc. of water during three minutes, the filtrate should be colorless (evidence of *complete carbonization*).

Remarks.—This is made by digesting animal charcoal with hydrochloric acid on a sand-bath for a period of eight hours, and, after straining the liquid, returning the charcoal to the vessel. The operation is repeated with a second amount of hydrochloric acid, and the digestion continued for two hours. Finally, the acid is drained off, the charcoal freed from all traces of acid by washing with water, and the product dried. The acid effects the solution of the bone-ash by converting it into soluble acid calcium phosphate, described in the manufacture of phosphorus (p. 404). Purified animal charcoal is an excellent decolorizer, and should always be used by the pharmacist in preference to the crude product.

The removing of color by means of purified animal charcoal is done by macerating the liquid with the charcoal for several hours, and finally filtering. It is well to remember that animal charcoal not only absorbs the color from a solution, but also absorbs some active plant principles, such as alkaloids and glucosides. Therefore, care should be taken to avoid the loss of active ingredients by too long contact with the charcoal.

Lamp-black is carbon produced by burning rosin, petroleum, or natural gas in a limited amount of air, and collecting the "soot" thus produced.

BORON

Symbol, B. Atomic weight, approximately 11

The word boron comes from *boraq*, which was the Arabic name for borax. The element was first isolated by Gay Lussac and Thenard in 1808.

Boron occurs in nature in the form of boric acid, sodium borate, and boracite, the latter being a magnesium borate. It is found in two allotropic forms, crystalline and amorphous. Its compounds with hydrogen and oxygen yield several acids, such as boric, metaboric, and pyroboric acids. Boron is not official, but it is found in official bodies—boric acid and borax.

ACIDUM BORICUM. Boric Acid.



It should contain not less than 99.8 per cent. of pure Boric Acid $[\text{B}(\text{OH})_3]$.

Transparent, colorless scales, of a somewhat pearly lustre, or six-sided, triclinic crystals, or a light, white, very fine powder, slightly unctuous to the touch; odorless, having a faintly bitter taste, and permanent in the air.

Boric Acid is soluble in 18 parts of water, 15.3 parts of alcohol, and 4.6 parts of glycerin at 25° C. (77° F.); in 3 parts of boiling water and 4.3 parts of boiling alcohol. The addition of hydrochloric acid decreases its solubility in water.

When heated to 100° C. (212° F.), Boric Acid loses water, forming metaboric acid (HBO_2), which slowly volatilizes at that temperature.

Heated to 160° C. (320° F.), it fuses to a glassy mass of tetraboric (or pyroboric) acid ($\text{H}_2\text{B}_4\text{O}_7$); at a higher temperature the fused mass swells up, loses all of its water, and becomes boron trioxide (B_2O_3), which fuses into a transparent, hygroscopic, non-volatile mass.

Boric Acid readily volatilizes from a boiling aqueous solution.

Tests for Identity.—Green flame. See Part V. Turns turmeric paper brown. See Part V.

Impurities.—Sulphates, chlorides, calcium, magnesium, heavy metals, arsenic, and not more than trace of iron. Details of each test in Part V.

Assay.—Details in Part V.

Remarks.—The formula of this compound, commonly called *boracic acid*, has already been discussed (p. 373), so here it is only necessary to emphasize the fact that boric acid is only true official ortho-acid.

Boric acid is found in the free form in lagoons in volcanic regions of Tuscany, the water being evaporated with the aid of the jets of steam emitted from the volcanic soil.

It is now made in large quantities in this country from native borax (p. 459) by treating it with hydrochloric acid, the equation being:



Boric acid is found in the form of crystalline micaceous scales, which possess a peculiar unctuous feeling. Boric acid is volatile, but more freely, however, in the vapor of water than if heated by itself. Boric acid is now popular in the form of an impalpable powder, and the preparation of this caused considerable annoyance on account of the slipperiness of the crystals, until it was found that trituration of boric acid in a warmed mortar or mill brings the desired results.

The two tests of boric acid are, first, the fact that an alcoholic solution burns with a flame enveloped with a green mantle, and, secondly, its property of changing yellow tumeric paper to a reddish brown.

SILICON

Symbol, Si. Atomic weight, approximately 28

The word silicon is derived from *silica*, or sand, which in turn is derived from the Latin *silex*, which means flint. The element was isolated by Berzelius in 1810. Silicon is the most abundant element on the earth's surface.

Thus, quartz, sand, opal, agate, and infusorial earth are more or less pure silicic oxide.

Silicon is another element appearing in two allotropic forms, crystalline and amorphous. None of the forms of silicon is of sufficient pharmaceutical interest to merit more than passing notice. Silicon, as shown on p. 369, yields one oxide, SiO_2 . The hypothetical combinations of this, with water, are silicic acids, H_2SiO_3 and H_4SiO_4 , and the salts of these acids are the ordinary silicates. Silicates of complex formulas, such as *orthoclase*, $\text{K}_2\text{AlSi}_6\text{O}_{16}$, are known.

Glass is a combination of the silicates of sodium or potassium with lead or calcium.

The test for soluble silicates is that they yield with acids a white gelatinous precipitate of orthosilicic acid, H_2SiO_4 .

The silicates other than those of the alkalis are insoluble in all acids, even the strongest, except hydrofluoric acid.

In order to obtain these silicates in soluble form it is necessary to fuse them with a mixture of sodium and potassium carbonates, whereby silicates of sodium and potassium are formed.

The only official preparation of silicon in the present pharmacopœia is kaolin. This will be considered among the compounds of aluminum (p. 539).

Liquor sodii silicatis, which was official in the pharmacopœia of 1890, is discussed on p. 474.

CHAPTER XXIV

THE ACIDS

AN acid is a substance which changes blue litmus-paper red, and combines with bases to form salts. The substances answering this definition are both inorganic and organic, and among the inorganic acids, as already mentioned, are found both binary or ternary compounds. The binary compounds are the four bodies, hydrochloric acid, HCl , hydrobromic acid, HBr , hydriodic acid, HI , and hydrofluoric acid, HFl . These four are called *haloid acids*, because derived from the four halogen elements. Three of these haloid acid are recognized by the pharmacopœia either alone or in pharmaceutic preparations.

Organic acids consist of carbon radicals combined with the group *carboxyl*, COOH . These will be discussed under their proper grouping in Part IV.

The inorganic acids are, with the exception of the haloid acids just mentioned, hydrates of the negative elements. Acids may be solids, liquids, or gases; in fact, all the members of this large group have little in common save their acid action on litmus-paper and their ability to form salts with the alkalis.

In this chapter we will discuss all the official inorganic acids except boric acid, chromic acid, and arsenous acid, the three being considered under the elements from which derived. All those considered in this chapter are liquids, and are commercially called the *heavy acids*, because their specific gravity is greater than water. These acids are usually in the concentrated form, and decidedly caustic. Hence in handling them great care must be taken to avoid bringing them in contact with delicate substances. If, in handling, they are spattered on the skin, they should be immediately washed off; if the area of burn is considerable, it should be covered with emollients of mildly alkaline character, such as lime liniment or sodium bicarbonate. Their active character prevents their ordinary storage in wood or metal, although it might be of interest to mention that sulphuric acid in concentrated form can be safely stored in iron tanks, that acid attacking iron only when diluted. In pharmacy, however, such acids must be handled entirely in glass or porcelain, and in purchasing quantities of the same they are usually

furnished in large glass containers boxed with straw, called *carboys*. In handling these carboys it is usually advisable to have trunnions on which they can be rocked (Fig. 216), though, with careful handling, this expense may be avoided.

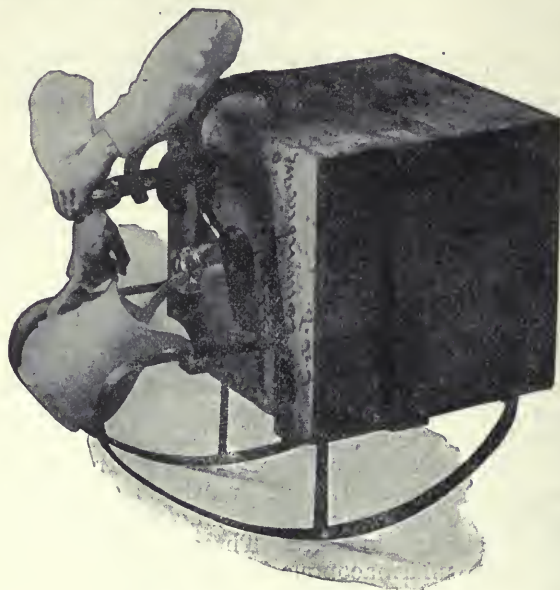


Fig. 216.—Carboy trunnion.

There is no uniformity in the commercial strength of the different strong acids, the usual custom being to furnish an acid of the greatest concentration feasible.

It is useful to memorize the following strengths and specific gravities:

	HCl	H ₂ SO ₄	HNO ₃
Percentage of absolute acid.....	31.9	92.5	68.00
Specific gravity (approximate).....	1.16	1.83	1.40

While the pharmacopœial concentrated acids vary in strength, the same standard demands that diluted acids contain 10 per cent. of absolute acid, except diluted acetic acid (6 per cent.) and diluted hydrocyanic acid (2 per cent.). A very convenient method of estimating the strength of acids is by their specific gravity. As an illustration, it will be noted above that the specific gravity of sulphuric acid is 1.83. Water, which is the only likely adulterant, has, of course, the specific gravity of 1.00, and if sulphuric acid is diluted with water, the specific gravity will fall to a point somewhere between 1.83 and 1.00. The only official liquid acid whose specific gravity is not a criterion of strength is acetic acid (see p. 672). The antidote for acids is mild alkali, such as calcined magnesia or lime liniment, this being a good illustration of a chemical antidote acting on an acid to form a neutral and inert salt. Too much emphasis cannot be placed on the word "mild," for while it is true that con-

centrated sodium hydrate or other strong alkali would form with the acid a neutral salt, yet the action of the same alkali is as dangerously caustic as is the acid for which it is intended as an antidote.

Most inorganic acids are prepared by the action of sulphuric acid on a salt of the acid which is desired. Sulphuric acid possesses a greater affinity for metals than most other acids, hence when a salt of the acid is treated with sulphuric acid, a sulphate is formed and the acid is liberated.

Reviewing the manufacture of official acids, we see how often this rule applies:

Hydrochloric acid,	made from	sodium chloride and sulphuric acid.
Hydrobromic	" "	potassium bromide and sulphuric acid.
Nitric	" "	sodium nitrate and sulphuric acid.
Chromic	" "	potassium dichromate and sulphuric acid.
Carbonic	" "	sodium bicarbonate and sulphuric acid.

Exceptions to this method are:

Phosphoric acid,	made by	oxidation of phosphorus with nitric acid.
Sulphurous	" "	reduction of sulphuric acid with carbon.

The officials treated in this chapter are:

Hydrochloric acid	containing 31.9 per cent. of absolute	HCl.
Diluted hydrochloric acid	" 10 "	" HCl.
Diluted hydrobromic acid	" 10 "	" HBr.
Diluted hydriodic acid	" 10 "	" HI
Syrup of hydriodic acid	" 1 "	" HI
Nitric acid	" 68 "	" HNO_3
Diluted nitric acid	" 10 "	" HNO_3 .
Nitrohydrochloric acid.		
Diluted nitrohydrochloric acid.		
Sulphuric acid	" 92.5 "	" H_2SO_4 .
Diluted sulphuric acid	" 10 "	" H_2SO_4 .
Aromatic sulphuric acid	} containing about 20	" H_2SO_4 .
Sulphurous acid		" SO_2 .
Phosphoric acid	" 85 "	" H_3PO_4 .
Diluted phosphoric acid	" 10 "	" H_3PO_4 .
Hypophosphorous acid	" 30 "	" $\text{H}_3\text{P}_2\text{O}_5$.
Diluted hypophosphorous acid	" 10 "	" $\text{H}_3\text{P}_2\text{O}_5$.

OFFICIAL ACIDS

ACIDUM HYDROCHLORICUM. Hydrochloric Acid.

A liquid composed of 31.9 per cent., by weight, of absolute Hydrochloric Acid [$\text{HCl} = 36.18$], and 68.1 per cent. of water. It should be kept in glass-stoppered bottles.

A colorless, fuming liquid, of a pungent odor, and an intensely acid taste; the fumes and odor disappear on diluting the Acid with 2 volumes of water.

Specific gravity: about 1.158 at 25°C . (77°F).

Miscible, in all proportions, with water or alcohol.

On distilling it, at first a stronger acid passes over, until, at 110°C . (230°F .), a liquid containing 20.13 per cent. of the absolute acid remains (specific gravity, about 1.098 at 25°C .), which distills unchanged, leaving no residue.

Hydrochloric Acid, even, when highly diluted, shows an intensely acid reaction with blue litmus-paper.

Heated with manganese dioxide it evolves chlorine.

With silver nitrate T.S. it yields a white, curdy precipitate, insoluble in nitric acid, but readily soluble in ammonia water.

If 10 Cc. of the Acid be evaporated from a platinum or porcelain dish, and dried at 110°C . (230°F .), no appreciable residue should remain (limit of *non-volatile impurities*).

Tests for Identity.—Tests for HCl and non-volatile impurities just given.

Impurities.—Non-volatile impurities (just given), bromine, iodine, free chlorine, arsenic, sulphuric acid, sulphates, sulphurous acid, and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This is an aqueous solution of hydrochloric acid gas, containing 31.9 per cent. of absolute acid, and having a specific gravity of 1.16. Its two synonyms—*spirit of sea-salt* and *muratic acid*—suggest its origin, the word *muratic* being derived from the Latin *murias*, which means brine. As mentioned above, it is prepared by distilling a mixture of salt (sodium chloride) and sulphuric acid.

ACIDUM HYDROCHLORICUM DILUTUM. Diluted Hydrochloric Acid.

Diluted Hydrochloric Acid should contain 10 per cent., by weight, of absolute Hydrochloric Acid [$\text{HCl} = 36.18$] and 90 per cent. of water. It should be kept in glass-stoppered bottles.

Recipe.—Hydrochloric Acid, one hundred grammes. 100 Gm.

Distilled Water, two hundred and nineteen grammes 219 Gm.

To make three hundred and nineteen grammes. . 319 Gm.

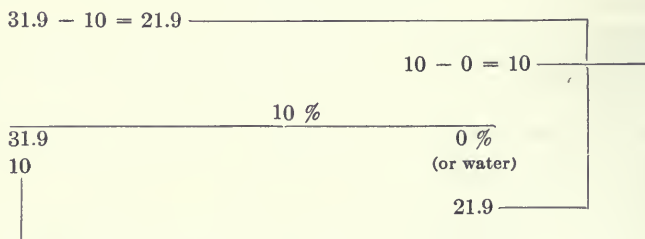
Mix them.

Specific gravity: about 1.049 at 25°C . (77°F).

It does not fume in the air and is without odor, but otherwise it should conform to the reactions and tests given under *Acidum Hydrochloricum*.

Assay.—Details in Part V.

Diluted hydrochloric acid is prepared by mixing 100 Gm. of hydrochloric acid (U. S. P.) with 219 Gm. of water. It will be noticed that the preparation strictly follows the rules of dilution performed by alligation. The problem is to reduce a 31.9 per cent. acid to a 10 per cent. acid. Following general rules of alligation, we get:



Summed up, we find that

10 Gm. of the official strong acid (31.9 per cent.)
and 21.9 Gm. of water (0 per cent.)
gives 31.9 Gm. of the diluted acid (10 per cent.).

Then, by proportion:

If to make 31.9 Gm. diluted acid we require 10 Gm. of strong acid; then to make 319 Gm. diluted acid we require $\frac{319}{31.9} \times 10$ or 100 Gm. stronger acid.

Or, expressed by equation:

$$\begin{array}{rcl} 31.9 & : & 10 \\ \times & \text{equals} & \frac{10 \times 319}{31.9} \end{array} \begin{array}{l} :: 319 : \times \\ \times 319 \\ \text{equals 100 Gm.} \end{array}$$

Hydrochloric acid, always in diluted form, is used in indigestion.

Dose.—Of the diluted acid, 1 Ce. (15 minims).

ACIDUM HYDROBROMICUM DILUTUM. Diluted Hydrobromic Acid.

A liquid composed of not less than 10 per cent., by weight, of absolute Hydrobromic Acid [$\text{HBr} = 80.36$] and about 90 per cent. of water. It should be kept in amber-colored, glass-stoppered bottles, protected from light.

A clear, colorless, and odorless liquid, having a strongly acid taste.

Specific gravity: about 1.076 at 25°C . (77°F .).

Miscible, in all proportions, with water or alcohol.

On distilling it, water and weak acid first pass over; when the temperature of 126°C . (258.8°F .) is reached, an acid of 48 per cent. remains, which may be distilled unchanged.

Diluted Hydrobromic Acid strongly reddens blue litmus-paper.

Silver nitrate T.S. causes a yellowish-white precipitate, which is insoluble in diluted nitric acid, but slowly soluble in an excess of stronger ammonia water, and readily soluble in solutions of sodium thiosulphate or potassium cyanide.

If copper sulphate T.S. be added to the Acid, a deep red color is produced upon the addition of sulphuric acid.

The Acid should not become colored on keeping.

Tests for Identity.—Given above.

Impurities.—Non-volatile impurities (test same as under hydrochloric acid). Sulphuric acid, barium, heavy metals, free bromine, iodine, arsenic, hydrochloric acid, and chlorides. Details in Part V.

Assay.—Details in Part V.

Remarks.—This official acid is a liquid containing 10 per cent. of absolute hydrogen bromide, and since an unstable chemical, should be dispensed as fresh as possible by the pharmacist. Hence efforts have been made to devise a simple method for its extemporaneous manufacture. The ordinary method of manufacture is by treatment of potassium bromide with sulphuric acid by the following reaction:



From this equation it will be seen that the product of the reaction other than hydrobromic acid is potassium sulphate. This dissolves in water with the hydrobromic acid, and to separate the acid from the sulphate solution the tedious process of distillation is necessary. This led investigators to attempt the separation of the second reaction product from the acid by precipitation, in which case the acid could be obtained by simple filtrations of the liquid from the precipitate. There are very few insoluble potassium salts, the best known being the bitartrate,—cream of tartar,—and this is utilized

in the Fothergill process of making hydrobromic acid. In this, instead of using sulphuric acid with potassium bromide, tartaric acid is employed, and, on standing, the bitartrate crystallizes out, the reaction being:



The above equation implies the molecular proportion of 119 Gm. potassium bromide and 150 Gm. tartaric acid in making 80 Gm. hydrobromic acid. Then to make 1000 Gm. 10 per cent. hydrobromic acid (containing 100 Gm. absolute acid) will require $\frac{100}{80} \times 119$, or 148 Gm. potassium bromide and $\frac{100}{80} \times 150$, or 187 Gm. tartaric acid.

These proportions do not agree with the original recipe of Dr. Wade, who planned an acid each dram of which would contain the equivalent of 10 grains of bromine; nor with Dr. Fothergill's recipe, which was a cumbersome one, adapted to the Imperial system of measures; nor with the simpler recipe of Professor Maisch.

The latter recipe consists of 80 grains of potassium bromide dissolved in 6 fluidrachms water; 100 grains tartaric acid dissolved in 2 fluidrachms water; the solutions mixed and then chilled, in order to facilitate the separation of the cream of tartar. The difficulty in striking an exact recipe is due to the uncertainty of the amount of cream of tartar precipitated, and in the metric recipe given above, the total amount of water used should be 900 Cc. Moreover, not more than 100 Cc. should be made at a time, since the product is not very stable.

It must be said that the product of this process is not absolutely pure acid; it always contains some potassium bromide and potassium bitartrate. Diluted hydrobromic acid is used as a sedative.

Dose.—4 Cc. (1 fluidrachm).

ACIDUM HYDRIODICUM DILUTUM. Diluted Hydiiodic Acid.

A solution of Hydiiodic Acid [HI = 126.9], containing not less than 10 per cent., by weight, of the Absolute Acid, and about 90 per cent. of water. It should be kept in amber-colored, glass-stoppered bottles, protected from the light.

<i>Recipe.</i> —Potassium Iodide, one hundred and thirty-five grammes.....	135.0 Gm.
Potassium Hypophosphite, ten grammes.....	10.0 Gm.
Tartaric Acid, one hundred and thirty-six and five-tenths grammes.....	136.5 Gm.
Distilled Water,	
Diluted Alcohol, each, a sufficient quantity,	
To make one thousand grammes. .	1000.0 Gm.

Dissolve the Potassium Salts in *two hundred and fifty cubic centimeters* of Distilled Water with the aid of heat, and the Tartaric Acid in *four hundred cubic centimeters* of Diluted Alcohol. Having poured the Solution of Tartaric Acid into a bottle of about one thousand cubic centimeters capacity, add the solution of Potassium salts and shake the mixture briskly. Place the bottle in a bath of ice-water for several hours, and having inserted a pledget of cotton tightly in the throat of a funnel, transfer the contents of the bottle to the funnel. When all the liquid has passed through, wash the bottle and crystalline precipitate with Diluted Alcohol in successive small portions until *one thousand grammes* of clear solution have been obtained. Evaporate the liquid at a moderate temperature, on a water-bath, until all of the Alcohol has been dissipated, and add sufficient Distilled Water to make the product weigh *one thousand grammes*.

A clear, colorless liquid, odorless, and having an acid taste.

Specific gravity: about 1.106 at 25° C. (77° F.).

Miscible, in all proportions, with water or alcohol.

On distilling Diluted Hydriodic Acid, water with some weak acid first passes over; at the temperature of 127° C. (206.6° F.) an acid of the strength of 57.5 per cent. distils over unchanged.

To blue litmus-paper it shows a strongly acid reaction.

Silver nitrate T.S. produces a yellow, curdy precipitate, insoluble in nitric acid, almost insoluble in ammonia water, but soluble in solutions of sodium thiosulphate and potassium cyanide.

If a few drops of ferric chloride T.S. or chlorine water be added to the Acid, diluted with twice its volume of water, iodine will be liberated and impart to the solution a reddish-brown color. On agitating the mixture with a few drops of chloroform, the latter will acquire a violet color.

The Acid should not become colored on keeping.

Diluted Hydriodic Acid, when evaporated to dryness on a bath of boiling water, and then heated to 115° C. (239° F.), should not leave more than 1 per cent. of residue.

Tests for Identity.—Just given.

Impurities.—Sulphuric acid, barium, heavy metals, and arsenic. Details in Part V.

Assay.—Details in Part V.

SYRUPUS ACIDI HYDRIODICI. Syrup of Hydriodic Acid.

A syrupy liquid containing about 1 per cent., by weight, of absolute Hydriodic Acid [HI = 126.9], or about 1.19 Gm. in 100 Cc.

<i>Recipe.</i> —Diluted Hydriodic Acid, one hundred grammes....	100 Gm.
Water, three hundred grammes.....	300 Gm.
Syrup, six hundred grammes.....	600 Gm.

To make one thousand grammes.. 1000 Gm.

Mix them.

A transparent, colorless, or not more than pale straw-colored liquid, odorless, and having a sweet and acidulous taste.

Specific gravity: about 1.190 at 25° C. (77° F.).

If a small portion of the Syrup be mixed with a little starch T.S., and a few drops of chlorine water then added, the liquid will acquire a deep blue color. Not more than a faint bluish tint should be produced in the Syrup by starch T.S. alone (limit of free iodine).

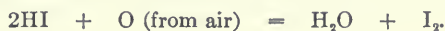
The addition of silver nitrate T.S. to a small portion of the Syrup diluted with four volumes of water produces a pale yellow precipitate, nearly insoluble in ammonia water.

Assay.—Details in Part V.

Remarks.—Hydriodic acid, as mentioned on p. 368, is the binary acid of iodine. It is official in the present pharmacopœia in the form of diluted (10 per cent.) acid and the syrup (containing 1 per cent. absolute hydriodic acid). The pharmacopœial method of the preparation of the diluted acid is a process devised by Dr. Buchanan, of Glasgow, and antedates the Fothergill-Wade process, which it closely resembles, the acid being liberated from potassium iodide by the addition of tartaric acid sufficient to precipitate potassium bitartrate, the equation being as follows:



The pharmacopœial recipe directs the addition of 1 per cent. of potassium hypophosphite, which is added to prevent, as far as possible, the separation of the iodine, which occurs when hydriodic acid is exposed to the air, the reaction being as follows:



Potassium hypophosphite is supposed to recombine the liberated iodine with hydrogen by the following reaction:



This decomposition, as well as that of ferrous iodide, has been a subject of considerable discussion of later years (see p. 551), recent investigations seeming to show that the accepted idea that the decomposition is due to the separation of free iodine is erroneous. Haussmann claims that spoiled specimens of the syrup contain no free iodine, and that the change noticed is the conversion of the cane-sugar into glucose, which change is accompanied by side reactions, which produce the brown color. His theory, however, as far as syrup of ferrous iodide (p. 551) is concerned, is not in accord with facts, since if the brown tint is due to caramelization of sugar, the syrup could not be restored to its green tint by the introduction of a few card teeth, as really occurs. Since iodine is liberated under oxidizing action of air, it is advisable to avoid combination of the official acid or the syrup prepared therefrom with oxidizing agents; hence potassium chlorate should not be dispensed with the syrup.

The pharmacopœia of 1880 directed a method for the manufacture of the syrup of hydriodic acid different from that of U. S. P. VIII., the process of 1880 being to treat water in which iodine was suspended with hydrogen sulphide gas, the equation being as follows:



As a matter of fact, this process leads to a purer form of hydriodic acid than does the process of the present pharmacopœia, since the attempt to precipitate all the potassium salt as potassium bitartrate is not entirely successful, inasmuch as the bitartrate is somewhat soluble. For this reason it is noticed that in the evaporation test for the acid given above 1 per cent. fixed residue is permitted.

Both the diluted acid and the syrup are used as alteratives.

Dose.—Of diluted acid, 0.5 Cc. (8 minims). Of syrup, 4 Cc. (1 fluidrachm).

ACIDUM NITRICUM. Nitric Acid.

A liquid composed of 68 per cent., by weight, of absolute Nitric Acid [HNO_3 or $\text{NO}_2\text{OH} = 62.57$] and 32 per cent. of water. It should be kept in glass-stoppered bottles.

A colorless, fuming liquid, very caustic and corrosive, and having a peculiar, somewhat suffocating odor.

Specific gravity: about 1.403 at 25° C. (77° F.).

It boils and is completely volatilized at 120.5° C. (248.9° F.).

It dissolves copper, mercury, silver, and other metals with evolution of red fumes, and stains woolen fabrics and animal tissues a bright yellow.

Heated with Indigo T.S., it discharges the blue color of the reagent.

Even when highly diluted, it shows an acid reaction upon blue litmus-paper.

Tests for Identity.—Given above.

Impurities.—Non-volatile impurities (test as in hydrochloric acid), arsenic, sulphuric acid, hydrochloric acid, heavy metals, iodine, bromine, iodic acid, and bromic acid. Detection of these given in Part V.

Assay.—Details in Part V.

ACIDUM NITRICUM DILUTUM. Diluted Nitric Acid.

Diluted Nitric Acid should contain 10 per cent., by weight, of absolute Nitric Acid [HNO_3 or $\text{NO}_2\text{OH} = 62.57$] and 90 per cent. of water.

Recipe.—Nitric Acid, one hundred grammes. 100 Gm.

Distilled Water, five hundred and eighty grammes.. 580 Gm.

To make six hundred and eighty grammes.. 680 Gm.

Mix them. Keep the product in dark amber-colored, glass-stoppered bottles. Specific gravity: about 1.054 at 25° C. (77° F.). It should respond to the reactions and tests given under *Acidum Nitricum*.

Assay.—Details in Part V.

Remarks.—Nitric acid is made by treating sodium nitrate with sulphuric acid and subsequent distillation. The official concentrated acid contains 68 per cent. of absolute acid, and has the specific gravity of 1.40. It was formerly largely dispensed under the synonym of *aqua fortis* (strong water), a synonym which, fortunately, is falling into disuse. The danger of the phrase is due to a confusion of it on prescriptions with *aqua fontana*, which means spring-water. Nitric acid is one of the most active of chemical substances, it being the most prominent oxidizing agent in general use (see p. 420). Great care should be taken in handling it to avoid contact with the skin or clothing, as it acts as a very strong caustic on both, either destroying the fabric or staining it yellow.

Diluted nitric acid is the above preparation diluted with water to a 10 per cent. preparation, the proportions following the rule of dilution by alligation. Nitric acid is given highly diluted as a tonic and astringent.

Dose.—Of official diluted acid, 2 Cc. (30 minims).

ACIDUM NITROHYDROCHLORICUM. Nitrohydrochloric Acid.

<i>Recipe</i> .—Nitric Acid, one hundred and eighty cubic centimeters	180 Cc.
Hydrochloric Acid, eight hundred and twenty cubic centimeters.....	820 Cc.
To make about one thousand cubic centimeters..	1000 Cc.

Mix the Acids in a capacious glass vessel, and, when effervescence has ceased, pour the product into dark amber-colored, glass-stoppered bottles, which should not be more than half filled, and which should be kept in a cool place.

A golden-yellow, fuming, and very corrosive liquid, having a strong odor of chlorine. Completely volatilized by heat. It readily dissolves gold leaf, and a drop of it, when added to potassium iodide T.S., liberates iodine.

ACIDUM NITROHYDROCHLORICUM DILUTUM. Diluted Nitrohydrochloric Acid.

<i>Recipe</i> .—Nitric Acid, forty cubic centimeters.....	40 Cc.
Hydrochloric Acid, one hundred and eighty-two cubic centimeters.....	182 Cc.
Distilled Water, seven hundred and seventy-eight cubic centimeters.....	778 Cc.
To make about one thousand cubic centimeters..	1000 Cc.

Mix the Acids in a capacious glass vessel, and, when effervescence has ceased, add the Distilled Water. Diluted Nitrohydrochloric Acid should be kept in dark amber-colored, glass-stoppered bottles, in a cool place. It should not be dispensed unless recently prepared.

A colorless or pale yellowish liquid, having a faint odor of chlorine and a very acid taste. It is completely volatilized by heat. When it is added to potassium iodide T.S., iodine is liberated.

Remarks.—Strong nitrohydrochloric acid is the *aqua regia* of the alchemists. Hydrochloric acid will not dissolve gold, neither will

nitric acid; but the two combined have this power, hence the alchemistic name, which, translated, means the kind of waters (*royal water*), since it dissolves gold, the king of metals. This special solvent power is due to presence, in the acid, of nitrosyl chloride, NOCl, and free chlorine.

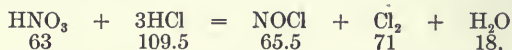
Aqua regia is made by the direct combination of nitric and hydrochloric acids, the reaction being as follows:



On figuring at the combining quantities used in the above equation and expressing same in terms of the official strength of the acid, it will be seen that the combining proportion is as follows:

Molecular Weight of Nitric Acid.	Molecular Weight of Hydrochloric Acid.	Molecular Weight of Nitrosyl Chloride.	Molecular Weight of Water.
H = 1	H = 1	N = 14	H ₂ O = 18
N = 14	Cl = 35.5	O = 16	
O ₃ = 48	HCl Mol. wt. = 36.5	Cl = 35.5	Molecular Weight of Chlorine.
HNO ₃ Mol. wt. = 63	3 HCl = 109.5	NOCl = 65.5	Cl ₂ = 71.

Repeating the equation:



we see that the proportions expressed are 63 Gm. absolute (100 per cent.) HNO₃ and 109.5 Gm. absolute (100 per cent.) HCl. Then, how much 68 per cent. HNO₃ and how much 31.9 per cent. HCl will be needed? Taking up the nitric acid problem first, we find it will require $\frac{100}{68} \times 63$ Gm., which equals 92.6 Gm. nitric acid U. S. P. (68 per cent.).

As to the hydrochloric acid, we will need:

$\frac{100}{31.9} \times 109.5$ Gm., which equals 343.2 Gm. hydrochloric acid U. S. P. (31.9 per cent.). That is, 92.6 Gm. official nitric acid, sp. gr. 1.403, and 343.2 Gm. of hydrochloric acid, sp. gr. 1.158, are required to fulfil molecular proportions. Then, how many cubic centimeters of each acid?

As to nitric acid:

$$\begin{array}{ll} 1 \text{ Cc. water} & = 1 \text{ Gm.} \\ 1 \text{ Cc. nitric acid} & = 1.403 \text{ Gm.} \end{array}$$

Hence, 92.6 Gm. nitric acid equals 92.6 divided by 1.403, which equals 66 Cc.

As to hydrochloric acid:

$$\begin{array}{ll} 1 \text{ Cc. water} & = 1 \text{ Gm.} \\ 1 \text{ Cc. hydrochloric acid} & = 1.158 \text{ Gm.} \end{array}$$

Hence, 343.2 Gm. hydrochloric acid equals 343.2 divided by 1.158, equals 296 + Cc. That is, nitrohydrochloric acid can be made by mixing 66 Cc. nitric acid U. S. P. with 296 Cc. hydrochloric acid U. S. P., or by union of 180 Cc. nitric acid with 810 Cc. hydrochloric acid:

$$66 : 296 :: 180 : \times \quad \times = \frac{296 \times 180}{66} = 810.$$

Comparison of these figures with the official recipe shows the latter provides slight excess of hydrochloric acid.

Diluted nitrohydrochloric acid is made by mixing 40 Cc. of nitric acid with 182 Cc. of hydrochloric acid, and after the reaction has ended, a sufficient quantity of water is added to make the official

product contain the equivalent of 3.8 per cent. absolute nitric acid and 6.7 per cent. absolute hydrochloric acid—that is, about 10 per cent. mixed acids. At first glance it would be seen that the same results could be obtained by blending together the same proportion of diluted nitric acid and diluted hydrochloric acid as is directed of the two concentrated acids, and this without the necessity of adding extra water. But in practice the preparation must never be made by combining the acids in diluted form, as then they would have a simple mixture of the two acids and not a solution of nitrosyl chloride and chlorine.

This recalls the fact that chemical reaction usually occurs best in fairly concentrated solution; that the more diluted the solution, the slower and less violent the reaction, for violence of reaction means simply the accomplishment of a large amount of work in a short space of time. Accordingly, if it is known that a reaction is apt to be very violent, its dangers may be lessened by a proper dilution of the ingredients. Another valuable controller of violent reactions is judicious cooling of the vessel.

Nitrohydrochloric acid is a hepatic stimulant when given in largely diluted doses. A prescription for liver troubles, formerly largely used, consisted of a mixture of fluidextract of taraxacum with the acid. When these two liquids are mixed, a violent effervescence occurs, due to the oxidation of some of the constituents of the fluidextract. In dispensing such preparations, therefore, care must be taken to avoid bottling until the effervescence ceases, as otherwise the liberated gases will cause an explosion of the bottle and the spattering of the acid mixture.

Dose.—Of strong acid, 0.2 Cc. (3 minims). Of official diluted acid, 1 Cc. (15 minims).

SULPHURIC ACID

(H_2SO_4)

This well-known substance has been known since the fifteenth century, it being a matter of record that Basil Valentine obtained it by heating ferrous sulphate; from this origin is the synonym, *oil of vitriol*, derived. The alchemist had become familiar with several chemicals of crystalline form and of different colors, such as the sulphates of zinc, copper, and iron. By reason of their similarity to bright pieces of glass these substances were termed respectively *white vitriol* (vitrous meaning glass), *blue vitriol*, and *green vitriol*. All three of these “glasses,” on being submitted to strong heat in a retort, yielded an oily liquid (sulphuric acid), which was, therefore, called oil of vitriol (or oil of glass).

Sulphuric acid is the most important manufactured chemical used, being the veritable foundation of modern chemical industry; therefore its process of manufacture must be clearly comprehended.

The first stage is burning sulphur in contact with air, when there is formed sulphurous oxide, SO_2 , whose suffocating odor is noticed in a burning sulphur match.

The second stage is the conversion of sulphurous oxide, SO_2 , into sulphuric oxide, SO_3 . This occurs by the process of oxidation—the

adding of oxygen to a body—a process not so simple as it at first seems. We cannot change SO_2 into SO_3 by simply mixing with oxygen. To accomplish this we have to employ an energetic compound like nitric acid, chromium trioxide, or potassium permanganate. Let us explain the reason for this.

The change from SO_2 to SO_3 is not simply an addition of one more atom of oxygen; there is another and very important difference—that of valence of sulphur:

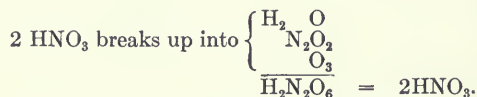
hex Sulphur in SO_2 is quadrivalent—has the valence IV—while in SO_3 it is *sex*valent—has the valence VI—and to change sulphur with four bonds to sulphur with six bonds requires energetic action. So oxidation is not merely adding oxygen to a body, but in inorganic chemistry it almost invariably means change in the valence of one of the elements acted on.

In the sulphuric acid process we have completed the second stage, save giving the equation showing the reaction, which is:



In writing reactions always balance the finished equation to see if the same number of atoms are found on either side of the sign of equality. This is most important, for otherwise the equation is defective. Let us now run over the reasoning whereby the balancing is done. Write down the equation given above, checking off as we go along. 3S, in the SO_2 groups: on the other side of the equation we have 3S in the SO_3 groups, check them. $3 \times 2\text{O}$ in the SO_2 groups and $2 \times 3\text{O}$ in the HNO_3 groups; that makes 12 O's in all. Let us look on the other side. $3 \times 3\text{O}$ in the SO_3 groups; 10 in the H_2O and 2O in N_2O_2 , they tally; so check them off. Then there are 2H in the HNO_3 groups, and on the other side there are 2H in H_2O ; check off. Lastly, there are 2N in the HNO_3 groups, and across the equality line there are 2N in N_2O_2 . When these are checked off, nothing unaccounted for remains on either side.

An interesting and useful point, especially in equation writing, is the amount of oxygen furnished by nitric acid in oxidation. 2HNO_3 give three atoms of oxygen, as is shown in the following equation:

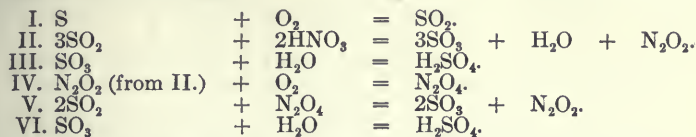


The third stage of the sulphuric acid reaction is very simple. The sulphuric oxide, SO_3 , comes in contact with steam, H_2O , forming sulphuric acid, H_2SO_4 .

In the second stage one of the products is N_2O_2 . This readily takes oxygen from the air, even in the lead chamber, changing from a colorless vapor to the red-brown fumes, N_2O_4 or 2NO_2 . This N_2O_4 is brought in contact with fresh quantities of sulphurous oxide, which it oxidizes into sulphuric oxide by the following reaction: $2\text{SO}_2 + \text{N}_2\text{O}_4 = \text{N}_2\text{O}_2 + 2\text{SO}_3$.

The resulting N_2O_2 is again converted into N_2O_4 , and thus the process is continuous, and a small quantity of nitric acid can be used

over and over again in the oxidization process. Let us sum up the reaction in its five stages:



From this stage on the reaction is continuous, employing I, V, IV, and VI. The appended sketch (Fig. 217) outlines the apparatus in which the reactions occur. The walls of the chamber are lined with lead; not because the metal is impervious to the acid, but because the lead sulphate formed is insoluble in water and sticks to the metal

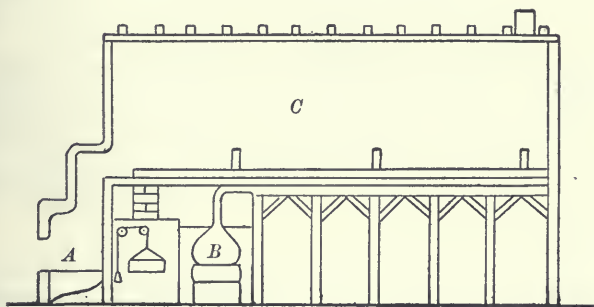


Fig. 217.—Lead-chamber (schematic): A, Sulphur furnace where reaction I takes place; B, retort where nitric acid is generated; C, lead-chamber where reactions II–VI occur.

as a protective covering. Were zinc, copper, or iron employed, the sulphates of the metal, being soluble in water, would be dissolved off by the steam, constantly leaving a fresh metallic surface to be attacked.

While a deviation from the subject directly under consideration, this is perhaps the best place to emphasize the necessity of learning to write and balance equations correctly—those short-hand representatives of chemical reactions found on almost every page from where first introduced (p. 368). As an illustration of how *not* to write equations may be cited the following remarkable reaction actually submitted the writer by one of his pupils:



How can a *sulphate* be formed when zinc and sodium hydrate are mixed? When we write, as we should—



we mean that if we combine 1 atom of zinc with 2 atoms sodium, 2 atoms oxygen, and 2 atoms hydrogen, we get, as the reaction products, 2 atoms hydrogen and also 2 atoms sodium, 1 atom zinc, and 2 atoms oxygen, and both besides must, of course, foot up the same.

To use a common comparison: if we take 4 apples, $\frac{1}{2}$ pound sugar, and 1 pound flour to make a pie, the finished pie will surely not contain 8 apples, 1 pound sugar and 10 pounds of flour. Neither will it contain 4 apples and a pound of baking soda. Use, in writing equations, the same common sense employed in discussing an ordinary apple-pie, remembering that a chemical reaction is merely a rearrangement of matter, and during such a change matter is neither created nor lost. While on the subject it may be well to emphasize the three varieties of reactions by giving equations expressing their character:

A *simple reaction* is one in which two elements combine to form a compound, thus: $H_2 + O = H_2O$.

A *simple decomposition* is a reaction in which an element acts on a compound in such manner as to break it up and then form a new compound, thus: $K + H_2O = KOH + H$.

A *double decomposition* is a reaction in which two compounds so act on each other as to form two new compounds, thus: $KOH + HCl = H_2O + KCl$.

To return to sulphuric acid, besides obtaining sulphurous oxide by burning sulphur in air, the same gas is obtained by the burning of iron pyrites (see p. 543), and in technic this substance is largely used for the production of sulphuric acid in place of sulphur. The objection to this so-called "pyrites acid" is that the native pyrites usually contain arsenic, and the acid obtained therefrom is frequently contaminated with this deleterious substance. Careful manufacturers, however, succeed in removing all traces of arsenic by treatment of the crude acid with hydrogen sulphide, the excess of which is removed from the acid by heating. According as to whether sulphur or iron pyrites is employed in the manufacture of the sulphuric acid, specially constructed furnaces are employed. It might here be said that the limitations of this work preclude entering into details of technic of the manufacture of all the chemicals here described; the student desiring such is referred to standard works on chemical technology found in Preface.

Of recent years, the *contact method* of manufacture of sulphuric acid has come into use. In this process the oxidation of sulphurous oxide to sulphuric oxide is accomplished by passing a mixture of the first-named oxide and of air over platinum heated to 400° – 450° C., thus avoiding the use of nitric acid—the air furnishing the required oxygen and the platinum bringing about the chemical action. The process is a commercial success, and is patented by a Swiss firm.

Sulphuric acid is the most important manufactured chemical known to the world, it being used in numberless branches of trade. An indication of its enormous consumption is the statement that in this country over one million tons are made each year.

Three forms of sulphuric acid are recognized by the pharmacopœia:

ACIDUM SULPHURICUM. Sulphuric Acid.

A liquid composed of not less than 92.5 per cent., by weight, of absolute Sulphuric Acid [H_2SO_4 or $SO_2(OH)_2 = 97.35$] and about 7.5 per cent. of water. It should be kept in glass-stoppered bottles.

A colorless liquid, of oily consistence, inodorous, and very caustic and corrosive. Specific gravity: not below 1.826 at 25° C. (77° F.).

Miscible, in all proportions, with water or alcohol, with evolution of much heat; the Acid should be added with great caution to the diluent.

It boils at 338° C. (640.4° F.). When heated on a platinum foil, it is vaporized, with the evolution of dense fumes, without leaving a residue.

Sulphuric Acid, even highly diluted, shows an acid reaction upon blue litmus-paper.

If Sulphuric Acid be dropped upon cane-sugar or wood, it chars them.

Diluted with 20 volumes of water, it yields with barium chloride T.S. a copious white precipitate, insoluble in hydrochloric acid.

If 2 Cc. of hydrochloric acid in which a fragment of sodium sulphite has been dissolved be carefully poured upon 2 Cc. of Sulphuric Acid contained in a test-tube, the zone of contact should not assume a pink or red color upon standing, nor should a precipitate form after heating (limit of *selenium*).

Test for Identity.—Given above.

Impurities.—Lead, nitric or nitrous acids, hydrochloric acid, heavy metals, non-volatile impurities, sulphurous acid, arsenic. (See Part V.) Selenium (see above).

Assay.—Details in Part V.

Remarks.—This contains 92.5 per cent. of the absolute acid, and has the specific gravity 1.826, which represents the greatest strength of acid convenient for manufacturing and handling. The pharmacopœia of 1880 recognized a 96 per cent. acid, but experience proved that the average commercial acid, even chemically pure, rarely attained this high degree of concentration, and since the 92.5 per cent. acid answered all practical requirements of pharmacy, that strength was substituted. This acid is a colorless liquid, of oily consistence, miscible in all proportions of alcohol and water, with the evolution of so much heat that mixing requires great caution.

ACIDUM SULPHURICUM DILUTUM. Diluted Sulphuric Acid.

Diluted Sulphuric Acid should contain not less than 10 per cent., by weight, of absolute Sulphuric Acid [H_2SO_4 or $\text{SO}_2(\text{OH})_2 = 97.35$] and about 90 per cent. of water.

Recipe.—Sulphuric Acid, one hundred grammes..... 100 Gm.

Distilled Water, eight hundred and twenty-five grammes..... 825 Gm.

To make nine hundred and twenty-five grammes.. 925 Gm.

Pour the Acid gradually, with constant stirring, into the Distilled Water. Keep the product in glass-stoppered bottles.

Specific gravity: about 1.067 at 25° C. (77° F.).

It should respond to the reactions and tests given under *Acidum Sulphuricum*.

Assay.—Details in Part V.

Remarks.—This diluted form contains 10 per cent. absolute H_2SO_4 . As mentioned above, the dilution of the strong acid with either alcohol or water must be performed with care, as great heat is evolved, frequently resulting in a spattering of the hot acid. A rule always to be observed in diluting sulphuric acid is that the acid should be poured in a fine stream into the water contained in a shallow dish. Never should water be poured into the acid, and rarely is it advisable to perform the dilution of the acid in anything but a shallow open dish.

Medical Properties.—Used like aromatic sulphuric acid. The concentrated is never used medicinally except after dilution.

Dose.—2 Cc. (30 minims).

ACIDUM SULPHURICUM AROMATICUM. Aromatic Sulphuric Acid.

Aromatic Sulphuric Acid should contain not less than 20 per cent., by weight, of absolute Sulphuric Acid [H_2SO_4 or $\text{SO}_2(\text{OH})_2 = 97.35$], partly in the form of ethyl-sulphuric acid.

Recipe.—Sulphuric Acid, one hundred and eleven cubic centimeters..... 111 Cc.

Tincture of Ginger, fifty cubic centimeters..... 50 Cc.

Oil of Cinnamon, one cubic centimeter..... 1 Cc.

Alcohol, a sufficient quantity,

To make one thousand cubic centimeters.. 1000 Cc.

Add the Sulphuric Acid gradually, and with great caution, to *seven hundred cubic centimeters* of Alcohol, and allow the mixture to cool. Then add to it the Tincture of Ginger and the Oil of Cinnamon, and afterward enough Alcohol to make the whole measure *one thousand cubic centimeters*. Keep the product in glass-stoppered bottles.

Specific gravity: about 0.933 at 25° C. (77° F.).

Assay.—Details in Part V.

Remarks.—This pharmaceutical is made by adding 111 Cc. of the concentrated acid to 700 Cc. alcohol, and when the mixture is cooled, there is added 50 Cc. of tincture of ginger, 1 Cc. of oil of cinnamon, and enough alcohol to make the finished product measure 1000 Cc.

At the first glance the above recipe would indicate a 10 per cent. acid, but when it is recalled that the 100 Cc. of sulphuric acid will weigh 184 Gm., while 100 Cc. alcohol weighs but 82 Gm., it will be seen that the strength by weight is about 20 per cent. This preparation is known as *elixir of vitriol*, and replaces the original recipe, which was popular for several hundred years. The preparation contains, besides sulphuric acid, some ethyl-sulphuric acid. (See p. 658.)

Aromatic sulphuric acid affords the favorite means of administration of the acid, its chief use being in the night-sweats of tuberculosis.

Dose.—1 Cc. (15 minims).

Nordhausen sulphuric acid is a form of acid obtained by distilling completely exsiccated ferrous sulphate or roasted iron pyrites in dry retorts. It is a thick liquid, consisting of a mixture of H_2SO_4 and SO_3 , and is, therefore, sometimes assigned the formula $H_2S_2O_7$. A semisolid modification now in commerce consists almost entirely of SO_3 .

ACIDUM SULPHUROSUM. Sulphurous Acid.

An aqueous solution containing not less than 6 per cent., by weight, of sulphur dioxide [SO_2 = 63.59] and about 94 per cent. of water.

Recipe.—Sulphuric Acid, *sixty cubic centimeters*. 60 Cc.
Charcoal, in coarse powder, *twenty grammes*. 20 Gm.
Distilled Water, *five hundred cubic centimeters*. 500 Cc.

Introduce the Charcoal into a glass flask having a capacity of about five hundred cubic centimeters, add the Acid, and mix them well. Connect the flask by means of bent glass tubing, about fifty centimeters in length, with a wash-bottle having a capacity of about two hundred cubic centimeters, containing about *fifty cubic centimeters* of water, so that the end of the inlet tube shall be below the surface of the water. Through the triply perforated rubber stopper of the wash-bottle pass a safety tube, which should reach nearly to the bottom of the bottle, and connect the latter, by means of glass tubing, with a bottle provided with a doubly perforated rubber stopper, having a capacity of about one thousand cubic centimeters, and containing *five hundred cubic centimeters* of well-cooled Distilled Water. The inlet tube should dip about twenty-five millimeters below the surface of the Distilled Water. By means of a second tube connect this bottle with another containing water, the end of the tube extending five centimeters below the surface. Having ascertained that all the connections are air-tight, apply a moderate heat to the flask containing the Sulphuric Acid and Charcoal, until the evolution of gas has nearly ceased, and, during the passage of the gas, keep the bottle containing the Distilled Water at or below 10° C. (50° F.) by surrounding it with cold water or ice. Assay a small portion of the Sulphurous Acid by the method given on p. 1004. Then add to the remainder sufficient Distilled Water to bring the product to the strength of 6.4 per cent., by weight, of sulphur dioxide. Finally, pour the Sulphurous Acid into dark amber-

colored, glass-stoppered bottles, which should be completely filled, and kept in a cool place, protected from light.

Owing to its rapid deterioration, Sulphurous Acid should be frequently assayed, and none should be dispensed if it fails to conform to the assay on p. 1004.

A colorless liquid, having the characteristic odor of burning sulphur, and an acid, sulphurous taste.

Specific gravity: not less than 1.028 at 25° C. (77° F.).

By heat it is completely volatilized. Blue litmus-paper moistened with the Acid is first reddened and afterward bleached.

On gently heating a few Cc. of the Acid in a test-tube, the gas evolved will blacken a strip of paper moistened with mercurous nitrate T.S., but will not affect one moistened with lead acetate T.S.

On mixing, in a test-tube, 1 Cc. of Sulphurous Acid with 5 Cc. of diluted hydrochloric acid, and adding a small piece of pure zinc, hydrogen sulphide gas will be evolved, which will blacken a strip of paper moistened with lead acetate T.S.

Tests for Identity.—Given above.

Impurities.—Sulphuric acid. Details given in Part V.

Assay.—Details in Part V.

The chemical is recognized by the pharmacopœia in the form of a saturated 6 per cent. aqueous solution. In common with all ternary acids, this body is derived from the corresponding oxide, sulphurous oxide, which we have already learned is SO_2 , and this combines with water, as shown in the following equation:



While theoretically all ternary acids are derived from the oxides by treatment with water, in the practical manufacture of ternary acids this method is not always evident. For instance, in the Markoe process of manufacture of phosphoric acid (p. 427) the formation of the oxide, while occurring, is not expressed in the equation. Sulphurous acid, however, is manufactured by the direct solution of sulphurous oxide gas in water, this gas being obtained by the reduction of sulphuric acid.

Two operations—oxidation, of the addition of oxygen to a body, and reduction, or the removal of oxygen from a substance—are very important operations in chemical work.

Substances capable of increasing the valence of an element at the same time it gives oxygen to the element are called oxidizing agents, and, on a preceding page, nitric acid, potassium permanganate, and chromic acid were cited as oxidizing agents. A reducing agent, on the other hand, takes oxygen from a body and reduces the valence of one of its elements. Carbon (charcoal), metallic sodium (chiefly as sodium alcoholate), and glucose are examples of reducing agents, and usually in inorganic chemistry carbon is employed. This is true in the manufacture of sulphurous acid, in which the reaction is as follows:



Both gases formed, sulphurous oxide and carbonic oxide, after washing,—after passing through a flask containing water, which will absorb such soluble impurities as sulphuric oxide,—are run into cooled distilled water, which dissolves the sulphurous oxide (forming corresponding acid), while most of the carbon dioxide passes off.

Since the pharmacopœial process affords a striking illustration of the apparatus required in the manufacture, washing, and collecting of gases, a picture of the apparatus is here given (Fig. 218). Sulphur dioxide and sulphurous acid are employed because of their reducing action, both abstracting oxygen from many substances with which they come in contact. It is because of this property that these bodies are used as bleaching agents and as disinfectants, and it is



Fig. 218.—Apparatus for manufacture of sulphurous acid U. S. P.

because of the latter action that the official acid is of value in medicine. Sulphurous acid is sometimes used internally as an antiferment. It soon loses the dissolved sulphurous oxide, hence should be dispensed only in the fresh state.

Dose.—2 Cc. (30 minims).

ACIDUM PHOSPHORICUM. Phosphoric Acid.

A liquid composed of 85 per cent., by weight, of absolute Orthophosphoric Acid [H_3PO_4 or $\text{PO}(\text{OH})_3 = 97.29$] and 15 per cent. of water. It should be kept in glass-stoppered bottles.

A colorless liquid of a syrupy consistence, without odor, and having a strongly acid taste.

Specific gravity: about 1.707 at 25° C. (77° F.).

Miscible, in all proportions, with water or alcohol.

When heated, the liquid loses water; at 200° C. (392° F.) it gradually begins to change to pyrophosphoric acid. At a still higher temperature it is converted into metaphosphoric acid, which volatilizes in dense fumes, or, on cooling, forms a transparent mass of glacial phosphoric acid.

The acid, even when largely diluted, has an acid reaction upon blue litmus-paper.

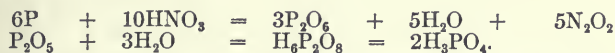
If a small portion of Phosphoric Acid be supersaturated with ammonia water, the addition of magnesium sulphate T.S. (or of magnesia mixture) produces a white, crystalline precipitate. If the precipitate be collected, washed, and dissolved in diluted acetic acid, the solution yields a yellow precipitate with silver nitrate T.S. (distinction from *metaphosphoric* or *pyrophosphoric acid*).

Test for Identity.—Given above.

Impurities.—Nitric acid, phosphorous acid, arsenic, phosphates, heavy metals, sulphuric acid, hydrochloric acid, metaphosphoric acid, pyrophosphoric acid. Details of these tests in Part V.

Assay.—Details in Part V.

Remarks.—Phosphoric acid is recognized by the pharmacopœia in the form of an 85 per cent. solution, the so-called “syrupy phosphoric acid.” It is made by the oxidation of phosphorous with nitric acid, a process accompanied by some danger, hence the recipe is omitted in the present pharmacopœia, since the chemical can be best and most cheaply prepared by the large manufacturer. The equation of the reaction is



Let us go over the reasoning employed in figuring out these quantities. We want to make P_2O_5 ; we need for this five atoms of oxygen. We have learned that 2HNO_3 yields three atoms of oxygen; three is not divisible into five; hence to make P_2O_5 will require the amount of oxygen from nitric acid corresponding to the least common multiple of three and five. This is fifteen. Fifteen atoms of oxygen are needed; 2HNO_3 yield three atoms oxygen; hence to furnish 15O will require $5 \times 2 \text{HNO}_3$ or 10HNO_3 .

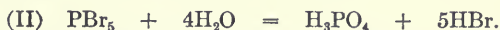
We are making P_2O_5 . One molecule of this contains five atoms of oxygen. We have fifteen atoms of oxygen; hence can make $3\text{P}_2\text{O}_5$; for $3 \times 5\text{O} = 15\text{O}$. Lastly, we see that in $3\text{P}_2\text{O}_5$ there are six phosphorus; hence we begin the equation with six atoms of phosphorus and ten molecules of nitric acid.

If to one molecule of P_2O_5 we add one molecule of H_2O , we get two molecules of metaphosphoric acid, HPO_3 . If, on the other hand, as above, we add three molecules H_2O to one molecule of P_2O_5 , we get two molecules of the official (so-called ortho-) phosphoric acid, H_3PO_4 .

A less dangerous method of manufacture than that just given, devised some thirty years since by Professor Markoe for the use of the retail pharmacist, consisted in treating phosphorus under the surface of water with bromine and a small quantity of iodine, and at the end of the reaction nitric acid is added. The reaction runs in three distinct stages. First, phosphorus and bromine unite to form phosphorus pentabromide, viz.:



This decomposes in the presence of water, with the formation of phosphoric acid and hydrobromic acid:



The nitric acid liberates bromine from hydrobromic acid:



The bromine here freed acts on another portion of the phosphorus, and the reactions I, II, and III are repeated in regular order as long as any phosphorus remains unoxidized.

It will be seen that the ultimate result of this process is the same as that of the first process, nitric acid being the real oxidizing agent in both cases. The function of the bromine in the second process is to lessen the violence of the reaction by gently bringing the nitric acid in contact with the phosphorus, insuring a gradual evolution of the gaseous nitrogen dioxide; for be it remembered that most explosions are due either to a sudden evolution or a sudden contraction of a gas. If the process is so planned to permit the evolution of a gas during several hours, no danger ensues, but if the same amount of gas is evolved in a few moments, a violent explosion occurs (p. 450).

In both processes the liquid remaining at completion of reaction is evaporated, partly to remove the last traces of nitric and nitrous acids (also bromine, in last process), and also to concentrate the product to the official strength. The last task is very tedious if carried out in an open evaporating dish, and in the writer's experience he has never succeeded in making an acid of pharmacopœial strength, the strongest he produced being about 65 per cent. Under the pharmacopœia of 1880, where a 50 per cent. acid was directed, the Markoe process was feasible for the retail pharmacist, but now that the pharmacopœia demands the more concentrated form, we are brought back to the original proposition that the pharmacist had better purchase his phosphoric acid. Much of the phosphoric acid of commerce contains arsenic, and the origin of this impurity is of interest as showing how persistently impurities may sometimes cling to chemicals, even though the original substance has undergone several changes. The arsenic in the phosphoric acid is due to arsenic-contaminated phosphorus. The phosphorus obtains it from arsenic-contaminated sulphuric acid, while the sulphuric acid contaminated with arsenic is usually that manufactured from iron pyrites, in which the arsenic originally occurred. It, therefore, behooves every careful pharmacist to test both his phosphoric acid and his sodium phosphate for arsenic by the tests prescribed in the pharmacopœia. (See p. 974.)

ACIDUM PHOSPHORICUM DILUTUM. Diluted Phosphoric Acid.

Diluted Phosphoric Acid should contain 10 per cent., by weight, of absolute Orthophosphoric Acid [H_3PO_4 or $\text{PO}(\text{OH})_3$ = 97.29] and 90 per cent. of water.

Recipe.—Phosphoric Acid, one hundred grammes..... 100 Gm.

Distilled Water, seven hundred and fifty grammes.. 750 Gm.

To make eight hundred and fifty grammes.. 850 Gm.

Mix them. Keep the product in glass-stoppered bottles.

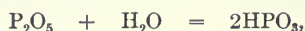
Specific gravity: about 1.057 at 25° C. (77° F.).

It corresponds in chemical properties and should conform to the reactions and tests given under *Acidum Phosphoricum*.

Assay.—Details in Part V.

Remarks.—This body represents the strong acid diluted with a sufficient quantity of water to make it contain 10 per cent. of absolute H_3PO_4 . These two acids are employed as nervous stimulants, although for such purpose that popular remedy, *acid phosphates*, is more frequently employed. A recipe for this preparation may be found in the National Formulary under the title of *liquor acidi phosphorici*. (See p. 407.)

It will be observed that the theoretic construction of the phosphoric acid is based on the addition of three molecules of water to one molecule of phosphoric oxide, this yielding H_3PO_4 —the so-called orthophosphoric acid. As explained on p. 373, this substance is not the true “ortho-” acid. If the same oxide is combined with one molecule of water, the following reaction occurs:



metaphosphoric acid being formed. This metaphosphoric acid is called *glacial phosphoric acid*, because on cooling it becomes a transparent, ice-like solid. A recipe for the *diluted metaphosphoric acid* may be found in the National Formulary.

Under this division mention may be made of *pyrophosphoric acid*, $\text{H}_4\text{P}_2\text{O}_7$. The composition of this body will be considered when discussing the official sodium pyrophosphate (p. 473), and mentions here made merely to call attention to the test distinguishing between the three forms, *ortho*-, *meta*-, and *pyro*-phosphoric acids, these three being also called respectively the tribasic, monobasic, and the tetrabasic phosphoric acids. (See p. 374.) Orthophosphoric acid does not coagulate albumin (white of egg), and forms a yellow precipitate of silver phosphate on the addition of silver nitrate solution. Metaphosphoric acid coagulates albumin, and gives a white precipitate with silver nitrate, while pyrophosphoric acid does not coagulate albumin, and gives a white precipitate with silver nitrate. Silver nitrate reactions also apply to the salts of these acids, but the albumin test can be used only with the free acids.

Dose.—2 Cc. (30 minims).

ACIDUM HYPOPHOSPHOROSUM. Hypophosphorous Acid.

A liquid composed of 30 per cent., by weight, of absolute Hypophosphorous Acid [$\text{PO.H}_2(\text{OH}) = 65.53$] and 70 per cent. of water. It should be kept in glass-stoppered bottles.

A colorless liquid, without odor, and having an acid taste.

Specific gravity: about 1.130 at 25° C. (77° F.).

Miscible, in all proportions, with water. Hypophosphorous Acid, even when largely diluted in water, has an acid reaction upon blue litmus-paper.

When heated in a porcelain dish, water evaporates, and the acid becomes more concentrated. On further heating between 130° and 140° C. (266° and 284° F.), it decomposes, forming hydrogen phosphide, which ignites, and phosphorous acid; the latter between 160° and 170° C. (320° and 338° F.) decomposes into hydrogen phosphide and phosphoric acid; the pasty residue finally reddens, ignites, and the last portions of unoxidized phosphorus burn out at a higher temperature.

The addition of silver nitrate T.S. to Hypophosphorous Acid, diluted with an equal volume of water, produces a black precipitate of metallic silver; the addition of mercuric chloride T.S., a white precipitate of mercurous chloride.

When the Acid is gently heated with copper sulphate T.S., a yellow precipitate forms, which rapidly assumes a reddish-brown color.

Tests for Identity.—Given above.

Impurities.—Barium, phosphoric, phosphorous, sulphuric, oxalic, and tartaric acids, heavy metals, potassium, arsenic. Details of each given in Part V.

Assay.—Details in Part V.

Remarks.—This new official contains 30 per cent. of absolute hypophosphorous acid. The usual method of manufacture is by a modification of the Fothergill process, potassium hypophosphite being treated with enough tartaric acid to precipitate all the potassium in the form of insoluble potassium bitartrate, the recipe given by the National Formulary being based on this reaction. Among other processes which have been suggested is the treatment of calcium hypophosphite with oxalic acid, in which case the following reaction occurs:



The calcium oxalate being then filtered from the solution of the acid. A third process is by treatment of barium hypophosphite with sulphuric acid, precipitated barium sulphate being removed by filtration.

The pharmacopœia writes the formula of hypophosphorous acid HPH_2O_2 . This is because it has but one replaceable hydrogen. Its composition has been the subject of considerable discussion, but now its graphic formula is supposed to be



The valence of phosphorus is five, just as in phosphoric acid. This is totally different from what we usually find in various acids of the same element, and constitutes one of the weak points in the valence theory.

ACIDUM HYPOPHOSPHOROSUM DILUTUM. Diluted Hypophosphorous Acid.

A liquid composed of 10 per cent., by weight, of absolute Hypophosphorous Acid [$\text{PO.H}_2(\text{OH}) = 65.53$] and 90 per cent. of water.

Recipe.—Hypophosphorous Acid, two hundred grammes.... 200 Gm.
Distilled Water, four hundred grammes..... 400 Gm.
To make six hundred grammes.. 600 Gm.

Mix them. Keep the product in well-stoppered bottles.

Specific gravity: about 1.042 at 25° C. (77° F.).

It should respond to the reactions and tests given under *Acidum Hypophosphorosum*.

Assay.—Details in Part V.

Remarks.—This 10 per cent. acid is directed in the present pharmacopœia to be made by diluting the official stronger acid. Diluted hypophosphorous acid is official because a constituent of the compound syrup of hypophosphites. It is rarely used alone, but when it is, its action is similar to that of the better-known hypophosphites of the alkalies.

Dose.—0.5 Cc. (8 minims).

CHAPTER XXV

THE METALS—POTASSIUM

THE elements thus far considered are grouped in the class which are called non-metals or metalloids. As already mentioned, however, a sharp distinction between the positive and the negative elements is a matter of considerable difficulty, and even so it is difficult to decide whether a given element is metallic or non-metallic. In choosing a

definition for the *metals*, about all that can be said is that those substances are metals which possess a metallic lustre and which are good conductors of electricity. Their oxides, when they dissolve in water, usually have an alkaline reaction, although several of the elements which we are now to consider will be found to possess oxides which are more acid than basic. It, therefore, resolves the definition to the one statement that metals are good conductors of electricity.

The alkaline metals are the group of elements, lithium, sodium, and potassium, to which is added the radical, ammonium (NH_4), it possessing properties so similar to the three metals just mentioned that its compounds are considered with them. It might be added, too, that the rare elements rubidium and caesium belong to this group, but since they have no pharmaceutical application, nothing need be said of them in this work.

These alkaline metals possess many characteristics in common. They all possess the valence 1, are soft and easily oxidizable substances, and are the only metals whose carbonates are soluble. This fact is used in analytic chemistry for separating the salts of these four metals from the other metals, as will be seen on p. 984.

POTASSIUM

Symbol, K. Atomic weight, approximately 39

This element is found in *carnallite*, which is a mixture of potassium and magnesium chlorides, and in *sylvite*, which is chiefly potassium chloride—these two ores are obtained from the mines of Stassfurt, Germany. Another source of potassium is *suint*, or the washings of wool. The freshly sheared wool is roughly cleaned by washing in water, the washings are then concentrated, and from them is obtained wool-fat by extraction with the proper solvent. In the residue is found potassium in sufficiently large amounts to be a commercial source of the element. Potassium carbonate is also extracted from the residues in the manufacture of beet-sugar (*vinasse*), while large amounts are obtained from ashes of wood by lixiviation.

Potassium was isolated by Sir Humphrey Davy in 1807 in his classic researches in electricity. He submitted potassa to electrolysis, placing a small lump on a piece of platinum foil, which was then connected with the negative pole of the battery. Bringing the positive pole in contact with the potassa the latter dissociated and small globules of metallic potassa were formed.

The electrolysis of potassium in Davy's day was too expensive a process for commercial application, and the metal was first produced commercially by Brunner by another process—by heating potassium carbonate with carbon in a retort to 1000°C ., when potassium and carbon monoxide were produced, as shown in the following equation:

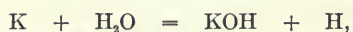


This process is accompanied with danger, inasmuch as carbon monoxide forms with potassium, as well as with several other metals (copper, for example), well-defined solid carbonyl derivatives. In the isolation of potassium the carbonyl, KCO , clogs up the delivery tube leading from the retort, and with the stoppage of this outlet

explosion of the retort occurs. This danger has been avoided by arranging the apparatus, whereby a plunger is made to fit in the delivery tube, and by passing this plunger up and down the obstructions were removed. It should have been stated that the delivery tube terminates in a vessel containing liquid petroleum or benzin, in order that the potassium will be condensed in a medium by which it is not affected, and which will protect it from atmospheric action.

Potassium is now being made on a large scale by electrolysis of potassium hydrate, the reduction in the cost of the production of electricity since Davy's day permitting the commercial application of Davy's process.

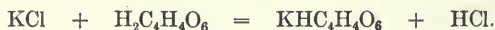
Potassium is a silvery metal, of the consistence of wax, hence it can be readily cut with a knife. A freshly cut surface, after exposure to air, becomes white and granular, due to the formation of the potassium carbonate. Metallic potassium combines so readily with oxygen that when exposed to the air the action is so energetic that the metal burns. Likewise, when the metallic potassium is thrown on the surface of water it combines with the same, forming potassium hydroxide, as shown in the following equation:



and the heat of the reaction is sufficient to cause an ignition of the hydrogen, which burns with explosive violence. Hence great care should be taken in the handling of potassium, and particular caution must be exercised in combining same with water. For this reason the metallic potassium is always kept under substances devoid of oxygen, such as benzin or petroleum.

The statements just given emphasize the care to be used in handling potassium. In weighing it one rapidly cuts from the piece the oxidized exterior and then presses the pure metal of the interior rapidly between filter-paper to remove the last traces of the liquid in which it has been kept. Thus it can be weighed upon a watch-glass, although in cases of large quantities it is best to weigh same under petroleum, leaving the drying of the metal until it has been weighed.

The tests for potassium are, first, the characteristic crystalline precipitate that potassium salts yield with platinic chloride. The separation of this precipitate is facilitated by the addition of a few drops of alcohol to the concentrated aqueous solution. Another test is the white, crystalline precipitate of cream of tartar, produced when a potassium salt is brought in contact with the proper quantity of tartaric acid, as shown in the following equation:



One of the most sensitive tests is the violet tint imparted to a colorless flame.

THE OFFICIAL COMPOUNDS AND PREPARATIONS OF POTASSIUM

Potassium acetate, 98 per cent. absolute $\text{KC}_2\text{H}_3\text{O}_2$.

Potassium bicarbonate, 99 per cent. absolute KHCO_3 .

Potassium bitartrate, 99 per cent. absolute $\text{KHC}_4\text{H}_4\text{O}_6$.

Potassium bromide, 97 per cent. absolute KBr .

Potassium carbonate, 98 per cent. absolute K_2CO_3 .

Potassium chlorate, 99 per cent. absolute KClO_3 .
 Lozenges of potassium chlorate. Pharmaceutic.
 Potassium citrate, 99 per cent. absolute $\text{K}_3\text{C}_6\text{H}_5\text{O}_7\text{H}_2\text{O}$.
 Effervescent potassium citrate contains 20 per cent. official citrate.
 Solution of potassium citrate contains 8 per cent. anhydrous citrate.
 Potassium cyanide, 95 per cent. absolute KCN .
 Potassium dichromate, 99 per cent. absolute $\text{K}_2\text{Cr}_2\text{O}_7$.
 Potassium and sodium tartrate, 99 per cent. absolute $\text{KNaC}_4\text{H}_4\text{O}_6\cdot 4\text{H}_2\text{O}$.
 Potassium ferrocyanide, 99 per cent. absolute $\text{K}_4\text{Fe}(\text{CN})_6\cdot 3\text{H}_2\text{O}$.
 Potassium hydroxide, 85 per cent. absolute KOH .
 Solution of potassium hydroxide contains 5 per cent. official hydroxide.
 Potassium hypophosphite, 98 per cent. absolute KPH_2O_2 .
 Potassium iodide, 99 per cent. absolute KI .
 Ointment of potassium iodide, containing 10 per cent. official potassium iodide.
 Potassium nitrate, 99 per cent. absolute KNO_3 .
 Potassium permanganate, 99 per cent. absolute KMnO_4 .
 Potassium sulphate, 99 per cent. absolute K_2SO_4 .

We might also add—

Iron and potassium tartrate (considered among the iron salts).
 Aluminum and potassium sulphate (considered among the aluminum salts).
 Solution of potassium arsenite (considered among the arsenic salts).

In going over the preparations of each metal we can usually note a certain salt of the metal that is used in making the other salts of the same metal. Thus, with the alkalies the carbonates are the salts usually employed. The salt thus selected as a "stock salt" is chosen for the most practical reasons. Among the deciding factors, cheapness and easy decomposition are the most important. Thus, with the alkalies, the chlorides are the most abundant salts, but since they are not easily decomposed, they are converted into carbonates, which are generally used. But besides these two factors other considerations come into play. Thus, in making potassium compounds the carbonate itself is not usually employed, because it is quite deliquescent and difficult to purify, therefore the bicarbonate, which occurs in handsome crystals, is selected. Hence, mere cheapness is not the absolute guide in the choice of material, but utility, average purity, and ease of action are among the other factors considered.

POTASSII HYDROXIDUM. Potassium Hydroxide.

$\text{KOH} = 55.74$

[POTASSA, PHARM. 1890]

It should contain not less than 85 per cent. of pure anhydrous Potassium Hydroxide, and not more than 2 per cent. of other inorganic substances, with the exception of water. It should be kept in well-stoppered bottles made of hard glass.

Dry, white flakes, fused masses, or in pencils, hard and brittle, showing a crystalline fracture; odorless, or having a faint odor of lye, and of a very acrid and caustic taste. *Great caution is necessary in tasting and handling it*, as it rapidly destroys organic tissues. Exposed to the air, it readily absorbs carbon dioxide and moisture, and deliquesces.

Soluble in about 0.4 part of water at 25°C . (77°F .), and in 2 parts of alcohol; very soluble in boiling water and in boiling alcohol; slightly soluble in ether.

When heated to about 530°C . (986°F .), Potassium Hydroxide melts to a clear, oily liquid, and at a bright red heat is volatilized unchanged. When introduced into a non-luminous flame, it imparts to it a violet color.

A solution of Potassium Hydroxide, even when greatly diluted, gives an intensely alkaline reaction with red litmus-paper.

The aqueous solution (1 in 20) should be perfectly clear and colorless.

A concentrated solution of Potassium Hydroxide, after acidulation with hydrochloric acid, yields a bright yellow precipitate with platinic chloride T.S.

A concentrated aqueous solution (1 in 10), when added to an excess of tartaric acid T.S., produces a white, crystalline precipitate, which redissolves when the Potassium Hydroxide is added in excess.

Tests for Identity.—Given above.

Impurities.—Heavy metals, no excess of carbonate. Details of these tests in Part V.

Assay.—Details in Part V.

Remarks.—The official chemical must contain at least 85 per cent. potassium hydroxide, a 100 per cent. specimen being almost impossible except when just solidified, as it rapidly absorbs water from the air.

Potassium hydroxide, usually made by the evaporation of a solution of potassa, the manufacture of which will be given below. Official potassa usually comes into the market in the form of cylindric sticks, about $\frac{1}{2}$ inch in diameter, these being made by melting the potassa and pouring into molds. The fusion of the potassa is an operation which must be carefully conducted, as the molten mass makes a very painful burn.

Potassa is very deliquescent, and therefore must be kept in well-stoppered bottles. It is a practice, on the first opening of a bottle of potassa, to seal it with a considerable quantity of wax, so that on opening at later periods an air-tight joint can be renewed by simply passing a hot spatula over the wax.

Potassa is considerably used in chemical manufacturing, as will be noted in the following pages. Its volumetric solution (see p. 993) is used for estimating the strength of acids. Medicinally, potassa is used externally as a caustic, the official sticks being brought in direct contact with the surface desired to be burned. Its action, however, is so severe that in modern medicine its use is comparatively rare. The internal administration of potassa is always in the form of the official solution.

It will be noted that the present pharmacopœia calls potassa "potassium hydroxide," and not potassium hydrate, the same rule being true of all metallic hydrates. (See Sodium Hydroxide, Ferric Hydroxide, etc.) This is in line with the modern chemical nomenclature, which attempts to limit the word hydrate to water of crystallization. (See p. 147.)

POTASSA CUM CALCE (U. S. P. 1890). Potassa with Lime.

The preparation, which was recognized by the pharmacopœia of 1890, represents the official potassa diluted with its own weight of freshly burned lime, the two being rubbed together in a warm iron mortar until a powder is formed. This preparation is supposed to be used as a caustic in preference to the pure potassa. It is never used internally.

LIQUOR POTASSII HYDROXIDI. Solution of Potassium Hydroxide.

[LIQUOR POTASSÆ, PHARM. 1890]

An aqueous solution, containing about 5 per cent. of Potassium Hydroxide [KOH = 55.74].

<i>Recipe.</i> —Potassium Hydroxide, sixty grammes.....	60 Gm.
Distilled Water, nine hundred and forty grammes..	940 Gm.
To make one thousand grammes..	1000 Gm.

Dissolve the Potassium Hydroxide in the Distilled Water.

The Potassium Hydroxide used in this process should be of the full strength and quality directed by the Pharmacopœia (85 per cent.). Potassium Hydroxide of

any other strength, however, may be used, if a proportionately larger or smaller quantity be taken, the proper amount for the above formula being ascertained by dividing 5100 by the percentage of absolute Potassium Hydroxide contained therein.

Solution of Potassium Hydroxide should be kept in bottles made of green glass, and provided with glass stoppers coated with paraffin or petrolatum.

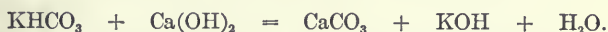
A clear, colorless liquid, odorless, having a very acrid and caustic taste, and a strongly alkaline reaction.

Specific gravity: about 1.046 at 25° C. (77° F.).

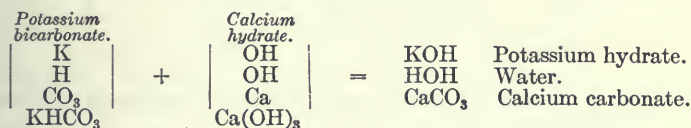
It should conform to the reactions and tests for an aqueous solution of potassium hydroxide given under *Potassii Hydroxidum*.

Assay.—Details in Part V.

Remarks.—This preparation, commonly called *solution of potassa*, is now directed to be made by the simple solution of stick caustic potash (potassium hydroxide of the present pharmacopœia) in water. The recipe of the U. S. P. 1890, given in detail in Part VII, consists of a double decomposition between potassium bicarbonate and calcium hydroxide, as shown in the following equation:



Let us make this, if possible, still clearer by graphic expression:



Note that in figuring out these processes it is the practical, rather than the theoretic, side that is considered. The compounds used are always the cheapest that will answer satisfactorily. Thus in the process just given, magnesium hydroxide could be used just as well as that of calcium, but it is not merely because it is no better for the purpose than slaked lime and it costs much more.

It will be noticed that the calcium is removed in the form of an insoluble calcium compound, leaving nothing in solution but potassium hydroxide, provided that the proper molecular proportions are employed.

Solution of potassa is a colorless liquid, having a very acrid caustic taste and strongly alkaline reaction. It is so strongly alkaline that it attacks the glass of the bottles in which it is kept, forming therein a precipitate of potassium silicate. For the same reason it causes the glass stopper to adhere to the neck of the bottle. Therefore, if the solution is kept in glass-stoppered bottles, the stoppers would be protected by being smeared with petrolatum.

For the purposes of the practical pharmacist the method of manufacture of the present pharmacopœia is better than the chemical process given above, as large quantities of the solution of potassa should not be kept on hand, owing to the fact that it rapidly deteriorates in strength, and the present official process affords an easy method of making up small quantities.

Solution of potassa is used as an antacid.

Dose.—1 Cc. (15 minims) largely diluted in water.

In cases of poisoning by potassa or its solution the antidote is

that used for all caustic alkalies, namely, a mild acid, such as vinegar or lemon-juice.

Note that *potassium* and *potassa* are not the same. Potassium is the element—the light alkali metal of the symbol K. Potassa, or caustic potash, is the hydroxide of potassium, its formula being KOH. Abandon the antiquated habit of saying “chlorate of potash,” “carbonate of potash,” and so on. Say “potassium chlorate,” or potassium carbonate,” and thus have labels printed, gradually educating the public. The question of label printing deserves more time than is usually given it. We take the labels as the printer sees fit to spell them, because, forsooth! they are cheap.

Shop labels should follow pharmacopœial changes, and in so doing it is best to have both the old and the new names on the label, as suggested below; for some conservative people would be suspicious were only the new name on the label. The suggested label is as follows:

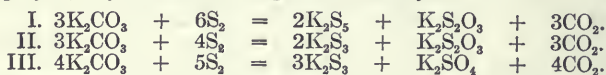
POTASSIUM CHLORATE.

(Chlorate of Potash.)

POTASSA SULPHURATA (U. S. P. 1890). Sulphurated Potassa.

This product, which was recognized by the former pharmacopœia, was dropped at the last revision. It is made by heating together potassium carbonate and sublimed sulphur in a covered crucible until the mass is in a state of perfect fusion.

According to the proportional quantities of the ingredients, and also the amount of heat employed, any of the following reactions may occur:

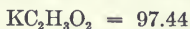


This shows that sulphurated potassa is not a definite chemical, but a mixture of the polysulphides, the sulphate, and the thiosulphate of potassium.

Sulphurated potassa, when freshly prepared, is in the form of irregular pieces, of a liver-brown color, and from the last fact it has been given the synonym *liver of sulphur*. On exposure to air, however, it absorbs moisture, oxygen, and carbon dioxide, changes to a greenish yellow, and eventually to a gray mass, the latter consisting of potassium carbonate, hyposulphite, and sulphate. It is needless to say that only the liver-colored preparation should be dispensed, and this necessity renders the preparation ineligible. The manufacture of it by the retail pharmacist is a matter of considerable difficulty and uncertainty, and yet if purchased in pound lots from the manufacturer, it becomes useless, or almost so, before a quarter has been used; moreover, there is no satisfactory method of preserving it.

Sulphurated potassa is used under the name of potassium sulphide as a laxative, being given in doses of 5 grains (0.3 Gm.).

POTASSII ACETAS. Potassium Acetate.



It should contain, when thoroughly dried, not less than 98 per cent. of pure Potassium Acetate $[\text{CH}_3\text{COOK}]$, and should be kept in well-stoppered bottles.

A white powder, or in crystalline masses of a satin-like lustre, odorless, and having a warming, saline taste. Very deliquescent on exposure to the air.

Soluble in 0.4 part of water and in 2 parts of alcohol at 25° C. (77° F.); with increasing temperature it becomes much more soluble in both liquids.

When heated to 292° C. (557.6° F.) the salt fuses. At a higher temperature it decomposes, blackens, and evolves vapors having an empyreumatic odor (an alliaceous odor would indicate the presence of *arsenic*), and, finally, if ignited on platinum, it leaves a white residue, which should be completely soluble in water.

The aqueous solution (1 in 20) is alkaline to red litmus-paper, but does not affect phenolphthalein T.S.

Tests for identity of potassium and acetic acid in Part V.

Impurities.—Heavy metals and arsenic. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by the treatment of potassium bicarbonate with acetic acid, as shown in the following equation:



The potassium acetate remains in the solution, while the carbon dioxide escapes in gaseous form, with copious effervescence. The solution of potassium acetate is then evaporated to dryness on a water-bath and immediately bottled.

Potassium acetate appears in the form of granules, odorless, and having a warm, saline taste. It is an extremely deliquescent salt, hence must always be kept in dry containers which are well sealed. It is given as a refrigerant and mild laxative.

Dose.—2 Gm. (30 grains).

Potassium arsenite, K_2HASO_3 or KAsO_2 , is official in the form of *Fowler's solution*, *liquor potassii arsenitis* (U. S. P.). This preparation will be discussed among the arsenic compounds.

POTASSII BICARBONAS. Potassium Bicarbonate.



It should contain not less than 99 per cent. of pure Potassium Bicarbonate [$\text{CO}(\text{OH})(\text{OK})$] and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms, or a colorless, odorless, granular powder, having a saline and slightly alkaline taste. Permanent in the air.

Soluble in about 3 parts of water at 25° C. (77° F.) and in 1.9 parts at 50° C. (122° F.). At a higher temperature the solution rapidly loses carbon dioxide, and, after being boiled, contains only potassium carbonate. Almost insoluble in alcohol.

The dry salt begins to lose carbon dioxide at 100° C. (212° F.), and this loss increases at a higher temperature, until, at a red heat, the salt has lost 30.96 per cent. of its original weight, leaving a residue of carbonate.

The concentrated aqueous solution of the salt is slightly alkaline to litmus-paper, but neutral to phenolphthalein T.S.

Tartaric acid T.S., added in excess to the concentrated aqueous solution, produces a white, crystalline precipitate.

Tests for Identity.—Given above.

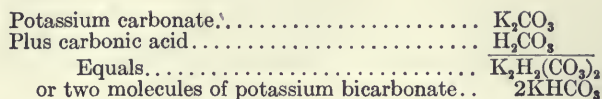
Impurities.—Excess of carbonate and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by exposing the carbonate to the action of carbonic acid gas.

In expressing the reaction by simple addition, it is easy to see why the product is called *bicarbonate*.

The reaction is:



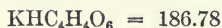
This shows that the bicarbonate really does contain twice as much carbonic acid as does the normal carbonate, a fact that is true of all salts bearing the prefix "bi." Formerly this process was carried on, suspending the carbonate in sacks over the fermenting vats of a brewery, and the crude bicarbonate thus obtained was placed on the market under the name of *saleratus*. At present the demand for *saleratus* is not large, and the carbonating is performed with carbon dioxide obtained from other sources, the resulting bicarbonate being purified by crystallization.

So many of our pharmaceutic chemicals depend upon water for their crystalline form that it is well to call attention to the fact that water is not essential in the formation of all crystals, potassium bicarbonate being an illustration of a chemical in well-defined crystals containing no water.

Potassium bicarbonate is an antacid. It is not often administered, its chief use being for the manufacture of other potassium compounds, as explained above. It might be noted that it is used as a carbonating agent in the manufacture of the solution of magnesium citrate.

Dose.—2 Gm. (50 grains).

POTASSII BITARTRAS. Potassium Bitartrate.



It should contain not less than 99 per cent. of pure Potassium Bitartrate [$\text{C}_2\text{H}_2(\text{OH})(\text{COOH})(\text{COOK})$] and should be kept in well-stoppered bottles.

Colorless or slightly opaque, rhombic crystals, or a white, somewhat gritty powder, odorless, and having a pleasant, acidulous taste. Permanent in the air.

Soluble in about 200 parts of water at 25° C. (77° F.) and in 16.7 parts of boiling water; very sparingly soluble in alcohol.

When a small portion of the salt is heated on platinum foil, it chars and emits inflammable vapors having the odor of burning sugar. At a higher temperature, with free access of air, the carbon of the black residue is oxidized, and a white, fused mass remains, which has an alkaline reaction and effervesces strongly with acids.

The aqueous solution of the salt has an acid reaction upon blue litmus-paper.

A solution of 0.5 Gm. of the salt in 3 Cc. of ammonia water should leave no insoluble residue (absence of *starch*, *kaolin*, *calcium phosphate*, and other *insoluble matter*).

Tests for identity of potassium and tartaric acid in Part V.

Impurities.—Starch, kaolin, calcium phosphate, and other insoluble matter; tests given above. Heavy metals, ammonia, and phosphates. Details in Part V.

Assay.—Details in Part V.

Remarks.—Potassium bitartrate (or *cream of tartar*) occurs in the form of a white, gritty powder; it is also seen in the form of small rhombic crystals, although this form is comparatively rare. It is the one official potassium compound which is sparingly soluble in water, one part requiring 200 parts of water at 25° C. to dissolve it.

The sparing solubility of cream of tartar is utilized in the manufacture of hydrobromic and other acids by the precipitation process. (See p. 413.) The fact just stated, however, that it does sparingly dissolve in water, shows us that hydrobromic acid made by the Fothergill process will be contaminated with bitartrate.

Cream of tartar is one of the most popular refrigerants and laxatives, being esteemed for sluggishness of the blood occurring during the spring and summer in warm climates. In such cases it is frequently administered in the form of lemonade, being combined with sugar and water, and possibly tamarinds, and the mixture taken in doses ranging from a wineglass to a cupful. Cream of tartar is a constituent of compound jalap powder.

Dose.—As diuretic, 2 Gm. (30 grains).

POTASSII BROMIDUM. Potassium Bromide.

$$\text{KBr} = 118.22$$

It should contain not less than 97 per cent. of pure Potassium Bromide and should be kept in well-stoppered bottles.

Colorless, or white, cubical crystals, or a granular powder; odorless and having a strongly saline taste. Permanent in the air.

Soluble in about 1.5 parts of water and in about 180 parts of alcohol at 25° C. (77° F.); in less than 1 part of boiling water and in 16 parts of boiling alcohol; also soluble in glycerin.

On heating the salt upon platinum foil it decrepitates; and at a temperature near 700° C. (1292° F.) it fuses without decomposing, and at a bright red heat volatilizes, communicating a violet color to the flame.

Its aqueous solution (1 in 20) is neutral, or has only a scarcely perceptible alkaline reaction upon litmus-paper.

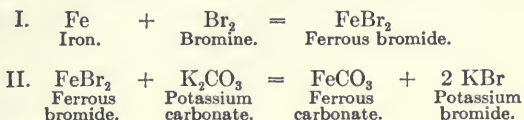
If 1 Gm. of Potassium Bromide be dissolved in 10 Cc. of water and 0.1 Cc. of tenth-normal sulphuric acid V.S. be added, no color should be produced by the subsequent addition of a drop of phenolphthalein T.S., even after heating (limit of *alkali*).

Tests for identity of potassium and bromide in Part V.

Impurities.—Bromate, iodides, heavy metals, and barium. Details of testing in Part V. Excess of alkali, see above.

Assay.—Details in Part V.

Remarks.—This valuable salt is prepared in two ways: The first process is the formation of ferrous bromide from iron and bromine, and the decomposition of this ferrous bromide by treatment with potassium carbonate, the two reactions being as follows:

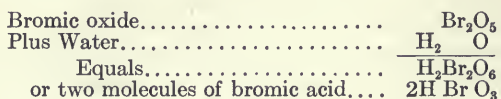


The double decomposition between ferrous bromide and potassium carbonate works admirably, as the insoluble ferrous carbonate precipitates, leaving only potassium bromide in solution. This is filtered and the bromide crystallized out.

The second process is a little more complicated, but highly instructive. In the first stage, potassium hydroxide is treated with bromine, thus:



We get potassium bromide, KBr , contaminated with potassium bromate, KBrO_3 , which is a salt of bromic acid, HBrO_3 , which is derived from bromic oxide, Br_2O_3 , in which compound bromine shows the valence v.



This bromate is an undesirable addition to the bromide, and must be converted into the latter. If we compare the formula of the bromide, KBr , with that of the bromate, KBrO_3 , we find the latter differs from the former only by having oxygen. This oxygen must be removed, and this is accomplished by the process of reduction, the reducing agent employed being carbon or charcoal, just as in the manufacture of sulphurous acid, the reaction being as follows:



Potassium bromide is used as a sedative and nervine, and is frequently administered in combination with chloral.

Dose.—1 Gm. (15 grains).

POTASSII CARBONAS. Potassium Carbonate.



It should contain, when thoroughly dried, not less than 98 per cent. of pure Potassium Carbonate $[\text{CO}(\text{OK})_2]$ and should be kept in well-stoppered bottles.

A white, granular powder, odorless, and having a strongly alkaline taste; very deliquescent.

Soluble in 0.91 part of water at 25°C . (77°F .) and in about 0.65 part of boiling water; insoluble in alcohol.

When heated to 130°C . (266°F .) the salt loses all the water which it may have retained or absorbed; at a bright red heat it melts, and at a white heat it volatilizes, communicating to a non-luminous flame a pure violet color.

No residue should be left on dissolving 1 Gm. of the salt in 20 Cc. of water (absence of earthy impurities).

Tests for identity of potassium and of carbonates in Part V.

Impurities.—Earthy impurities (given above), heavy metals and nitrate. Details of last two tests in Part V.

Assay.—Details in Part V.

Remarks.—Potassium carbonate is known by several synonyms, among others, *salt of tartar*. It is called this because it can be made by heating cream of tartar, the reaction being:



The other synonyms are *pearl-ash* and *salt of wormwood*, the latter name being derived from the fact that a variety of potassium carbonate, which was well esteemed some centuries ago, was made from the ashes of wormwood.

As already mentioned (p. 431), potassium carbonate is one of the chief sources of the element, and large quantities of the crude salt are obtained from suint and from ashes. At the same time, however, considerable is manufactured by the same process used in sodium

carbonate, and this process will be considered under making sodium carbonate (p. 461).

Potassium carbonate is a white, granular powder; it is very deliquescent, and hence should, like potassium acetate, be kept in well-stoppered bottles. It is used as an antacid, although its action is rather too irritating for general use, sodium bicarbonate being preferred. Taken in large quantities it has caustic action, and as an antidote, a mild acid should be used.

Dose.—1 Gm. (15 grains).

Potassium carbonate is used considerably in some sections as an alkaline hair-wash, the object being to prevent blond hair from turning dark.

For this purpose it is of little value, and is, moreover, decidedly detrimental to the health of the hair.

POTASSII CHLORAS. Potassium Chlorate.



It should contain not less than 99 per cent. of pure Potassium Chlorate [ClO_2 , OK] and should be kept in well-stoppered bottles. *Great caution should be observed in handling it*, as dangerous explosions are liable to occur when it is heated or subjected to concussion or trituration with organic substances (cork, tannic acid, sugar, etc.), or with sulphur, antimony sulphide, phosphorus, or other easily oxidizable substances.

Colorless, lustrous, monoclinic prisms or plates, or a white, granular powder; odorless and having a cooling, characteristic taste. Permanent in the air.

Soluble in 16 parts of water at 25°C . (77°F .) and in 1.7 parts of boiling water; insoluble in absolute alcohol and but slightly soluble in diluted alcohol.

At 334°C . (633.2°F .) the salt fuses, and above 352°C . (665.6°F .) it is decomposed into oxygen and potassium perchlorate; above 400°C . (752°F .) all of its oxygen is liberated, and a white residue of potassium chloride remains, amounting to 60.8 per cent. of the pure Chlorate employed. This residue is readily soluble in water, and the solution yields a white, curdy precipitate with silver nitrate T.S.

The aqueous solution of the salt (1 in 20) is neutral to litmus-paper.

Tests for identity of potassium and chlorates in Part V.

Impurities.—Heavy metals, nitrates, and nitrites. Details of these tests in Part V.

Remarks.—The formula of potassium chlorate is KClO_3 , from chloric acid, HClO_3 , from chloric oxide, Cl_2O_5 , the graphic formula which was given on p. 365.

The formula of potassium chlorate, KClO_3 , is analogous to KBrO_3 , potassium bromate, and the chlorate can be made by passing chlorine into potassium hydroxide by a reaction similar to that occurring in the manufacture of potassium bromide:



But by this method, from six molecules of potassium hydroxide only one molecule of potassium chlorate is obtained, the remaining five atoms of potassium going to waste as potassium chloride, and a better method of manufacture is from chlorinated lime. This body, on being boiled with water, decomposes into calcium chloride and calcium chlorate by the following reaction:



The calcium chlorate solution is then treated with potassium chloride, when the following reaction occurs:



The resulting calcium chloride is very soluble in water, and remains in solution, while the sparingly soluble potassium chlorate crystallizes out.

Potassium chlorate is now largely made by electrolysis.

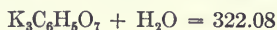
Potassium chlorate occurs in monoclinic crystals or plates or as a white powder, soluble in 16.7 parts of water at 15° C. and 1.7 parts of boiling water. Its solubility is worth more than passing notice, inasmuch as it is the custom of many drug-stores to keep on hand a stock solution of potassium chlorate which is labeled a half-drachm to the fluidounce. If by half-drachm 30 grains is intended, such a solution is impossible at ordinary temperatures, inasmuch as only 27 grains of the salt will dissolve in an ounce of cold water. Nor is it wise for the operator to try to coax the remaining potassium chlorate to go into solution by warming the mixture. A solution will ensue, but on cooling not only are the extra 3 grains crystallized out, but practically the entire salt separates in the bottom of the vessel in a crystallized mass. Physicians, not aware of the exact solubility, sometimes prescribe 30 grains of potassium chlorate to the ounce of solution, and intend same for gargle. If this prescription is dispensed with the excess of the salt remaining in the mixture, the undissolved salt will act as irritant to the inflamed mucous membrane which it is intended to relieve. Hence the careful pharmacist always dispenses a clear, filtered solution of potassium chlorate when intended for gargle.

Potassium chlorate is a powerful oxidizing agent, and for this reason combines with many easily oxidizable substances, such as sulphur, antimony sulphide, phosphorus, starch, sugar, so energetically as to produce explosions; hence the pharmacopœial warning that this salt should not be triturated with any of the above substances. When called upon to mix such substances as potassium chlorate and sugar, they should first be powdered separately and then mixed together with a bone spatula on a piece of paper.

Potassium chlorate is a mild antiseptic, this property being due to its oxidizing action. Its chief application for this purpose is in cases of sore throat, for which it is much esteemed. Its indiscriminate use, however, for this purpose, especially in the form of lozenges and compressed tablets, is not advisable, for investigation shows that it has an irritating action on the bladder, and therefore should not be swallowed in large quantities.

Dose.—250 milligrammes (4 grains).

POTASSII CITRAS. Potassium Citrate.



It should contain not less than 99 per cent. of pure Potassium Citrate $[\text{C}_3\text{H}_4(\text{OH})(\text{COOK})_3 + \text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

Transparent, prismatic crystals, or a white, granular powder, odorless, and having a cooling, saline taste. Deliquescent when exposed to the air.

Soluble in about 0.5 part of water at 25° C. (77° F.) and very soluble in boiling water; sparingly soluble in alcohol.

When heated above 100° C. (212° F.) the salt begins to lose water; at 200° C. (392° F.) the water of crystallization (5.55 per cent.) is completely lost. At 230° C. (446° F.) the salt begins to decompose, turns brown, and at a higher temperature carbonizes and emits inflammable gases which have a very pungent, acid odor. At a red heat a blackened mass of potassium carbonate and carbon is left, which has an alkaline reaction, and strongly effervesces with acids.

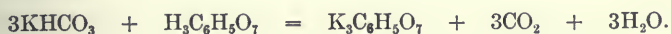
The aqueous solution of the salt is alkaline to red litmus-paper, but does not affect phenolphthalein.

Tests for identity of potassium and of citrate in Part V.

Impurities.—Heavy metals, tartrate. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by treatment of potassium bicarbonate with citric acid, as shown in the following equation:



As mentioned above, the bicarbonate is used in preference to the carbonates in the manufacture of most of the potassium salts, and in this case the bicarbonate is absolutely necessary, the citrate made from the carbonate being quite unsatisfactory.

The potassium citrate occurs in transparent prismatic crystals or in white granular powder, both forms being rather deliquescent. It is used as a refrigerant.

Dose.—1 Gm. (15 grains).

POTASSII CITRAS EFFERVESCENS. Effervescent Potassium Citrate.

<i>Recipe.</i> —Potassium Citrate, two hundred grammes.....	200 Gm.
Sodium Bicarbonate, dried and powdered, four hundred and seventy-seven grammes.....	477 Gm.
Tartaric Acid, dried and powdered, two hundred and fifty-two grammes.....	252 Gm.
Citric Acid, uneffloresced crystals, one hundred and sixty-two grammes.....	162 Gm.
To make about one thousand grammes..	1000 Gm.

Dry the Potassium Citrate on a water-bath until it ceases to lose weight; after powdering the dried salt, mix it intimately with the powdered Citric Acid and Tartaric Acid, then thoroughly incorporate the Sodium Bicarbonate. Place the mixed powders on a plate of glass or in a suitable dish, in an oven heated between 93° and 104° C. (199.4° and 219.2° F.).

When the mixture, by the aid of careful manipulation with a wooden spatula, has acquired a moist consistence, rub it through a No. 6 tinned-iron sieve, and dry the granules at a temperature not exceeding 54° C. (129.2° F.). Keep the product in well-stoppered bottles.

Remarks.—This is a granular salt, made by mixing powdered citric acid, potassium bicarbonate, and sugar together in a warm mortar, drying the resulting mixture by heating it rapidly in a chamber at not exceeding 120° C., and then powdering the mass and transferring to well-stoppered bottles.

The granular effervescent salts have been considered on p. 149, so here it suffices to say that this preparation is supposed to contain

and potassium bicarbonate in uncombined form, but that with water, these two chemicals combine, with liberation of carbonic oxide gas and copious effervescence, making the mixture palatable.

Dose.—4 Gm. (60 grains).

LIQUOR POTASSII CITRATIS. Solution of Potassium Citrate.

An aqueous liquid, containing in solution not less than 8 per cent. of anhydrous Potassium Citrate $[C_3H_4(OH)(COOK)_3=304.2]$, with small amounts of citric and carbonic acids.

<i>Recipe</i> .—Potassium Bicarbonate, eight grammes.....	8 Gm.
Citric Acid, six grammes.....	6 Gm.
Distilled Water, a sufficient quantity,	
To make one hundred cubic centimeters..	100 Cc.

Dissolve the Potassium Bicarbonate and the Citric Acid, each, in *forty cubic centimeters* of Distilled Water. Filter the solutions separately, and wash the filters with enough Distilled Water to obtain, in each case, *fifty cubic centimeters*. Finally, mix the two solutions, and, when effervescence has nearly ceased, transfer the liquid to a bottle. This preparation should be freshly made when wanted.

A clear, colorless liquid, odorless, having a mildly saline taste and a slightly acid reaction.

It should conform to the reactions and tests for an aqueous solution of the salt given under *Potassii Citras*.

Assay.—Details in Part V.

Remarks.—This preparation is made by dissolving separate solutions of potassium bicarbonate and citric acid, the quantity of the chemicals in each being so adjusted that when equal volumes of the two solutions are mixed, slightly acidulated potassium citrate will result. It is assumed that the pharmacist will not blend the two solutions until an order for the finished solution is received, and in this way the solution will be dispensed with sufficient effervescence remaining to make the liquid more palatable. The exact amount of “sparkle” is a matter of pharmaceutical judgment, for if the mixed liquid is quickly poured into a bottle and is tightly corked, there is danger of sufficient gas being evolved to cause an explosion of the flask in the hands of the purchaser.

The pharmacopœia of 1890 gave as a synonym of this solution *mistura potassii citratis*, because this preparation is intended to replace the mixture of potassium citrate (*neutral mixture*) of the pharmacopœia of 1880. The product of 1880 was much more palatable than the present official solution, because instead of citric acid, fresh lemon-juice was employed.

Potassium citrate is a mild refrigerant, and when dispensed in the effervescent form, constitutes a refreshing draft for fever patients.

A modification of the preparation quite popular in French countries is *potion de Riviere*. This is made by preparing two solutions, one consisting of citric acid, 2 parts; water, 50 parts; syrup, 15 parts; the other consisting of potassium bicarbonate, 2 parts; water, 50 parts; syrup, 15 parts; dispensing the two solutions in separate bottles, with directions to patient to mix equal parts and drink while effervescing.

Dose.—Of official solution: 16 Cc. (4 fluidrachms).

POTASSII CYANIDUM. Potassium Cyanide.

KCN = 64.70

It should contain not less than 95 per cent. of pure Potassium Cyanide and should be kept in well-stoppered bottles.

White, opaque, amorphous pieces, or a white, granular powder, odorless when perfectly dry; deliquescent in the air and exhaling the odor of hydrocyanic acid. *Great caution should be used in tasting and handling this salt.*

Soluble in about 2 parts of water at 25° C. (77° F.). Boiling water dissolves its own weight of the salt, but rapidly decomposes it. In alcohol it is but sparingly soluble.

At a low red heat the salt fuses.

Its aqueous solution (1 in 20) has a strongly alkaline reaction upon red litmus-paper and emits the odor of hydrocyanic acid.

Tests for identity of potassium and cyanides in Part V.

Impurities.—Excess of carbonate, ferrocyanide, sulphocyanate. Details in Part V.

Assay.—Details in Part V.

Remarks.—This official is made by heating potassium ferrocyanide with potassium carbonate in a red-hot crucible, when the following reaction ensues:



It will be seen from this reaction that the product will be contaminated with potassium cyanate, KOCN (p. 745). If a perfectly pure product is desired, it is made by passing hydrocyanic acid gas into an alcoholic solution of potassium hydroxide.

Potassium cyanide is now being made by an electric process. The demand for this chemical has greatly increased in late years, it being very largely used in metallurgic processes, the extraction of gold and silver from ores, etc.

Potassium cyanide occurs in white, irregular lumps, or as a white, granular powder, usually exhaling the odor of hydrocyanic acid, though odorless when perfectly dry. It is rather deliquescent, and therefore should be kept in well-stoppered bottles.

The pharmacopœia, in mentioning the tests, warns the observer of the great care required, inasmuch as the cyanide is a deadly poison.

When boiled with water, potassium cyanide splits up into potassium formate (p. 745) and ammonia, as shown in the following equation:



For this reason solutions of the cyanide should never be heated.

In medicine, potassium cyanide affords a convenient way of obtaining the medicinal action of hydrocyanic acid, and a prescription calling for potassium cyanide and citric acid was formerly considerably used.

Dose.—10 milligrammes ($\frac{1}{3}$ grain).

POTASSII DICHROMAS. Potassium Dichromate.K₂Cr₂O₇ = 292.28

[POTASSII BICHROMAS, PHARM. 1890]

It should contain not less than 99 per cent. of pure Potassium Dichromate [Cr₂O₇(OK)₂] and should be kept in well-stoppered bottles.

Large, orange-red, transparent, triclinic prisms, or four-sided tabular crystals, odorless, and having an acidulous, metallic taste. Permanent in the air.

Soluble in about 9 parts of water at 25° C. (77° F.) and in 1.5 parts of boiling water; insoluble in alcohol.

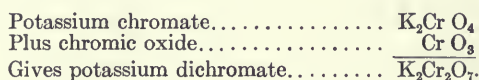
The salt fuses below a red heat, without loss of weight, forming a dark-brown liquid. At a white heat it evolves oxygen and leaves a residue of neutral potassium chromate and green chromic oxide.

The aqueous solution of Potassium Dichromate (1 in 20) has an acid reaction upon blue litmus-paper.

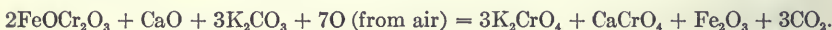
Tests for Identity.—See Potassium and Chromium Trioxide in Part V.

Remarks.—It will be noted that this chemical contains no hydrogen whatever, and in this case the rule laid down, that an acid salt is one in which all the hydrogen of the acid is not replaced by a metal, fails utterly. It is just such exceptions and variations in the nomenclature of chemistry that make its study difficult.

Let us dissect this compound, potassium dichromate, $K_2Cr_2O_7$. Its sister compound is potassium chromate, K_2CrO_4 , which is derived from chromic acid, H_2CrO_4 , which comes from chromic oxide, CrO_3 , a body analogous to SO_3 .



This compound is made from chromic iron ore, a mineral formerly mined in considerable quantities in Pennsylvania and Maryland, but now practically all imported from Europe. This ore is roasted with potassium carbonate and lime in presence of air, when the potassium chromate is formed by the following reaction:



This potassium chromate is then converted into potassium dichromate by treatment with sulphuric acid, as shown in the following equation:

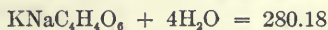


The calcium chromate of the first reaction is treated with potassium sulphate, and is thus converted into potassium chromate, which is changed to dichromate by use of sulphuric acid.

Dichromate of potassium occurs in orange-yellow crystals, which are soluble in 9 parts of water and in $1\frac{1}{2}$ parts of boiling water. Its chief use in pharmacy is as an oxidizing agent, for when treated with sulphuric acid it splits into chromic acid, one of the best oxidizing agents. It will be recalled that the oxidizing action of the dichromate mixture was suggested as a way of cleaning mortars. (See p. 114.) The dichromate mixture is also used as a battery fluid, a recipe for which will be found in the National Formulary under the name *Liquor Electropæicus*. It is rarely used in medicine; if so, it is employed as a remedy in syphilis.

Dose.—10 milligrammes ($\frac{1}{4}$ grain).

If an excessive dose is taken, the antidote would be a mild alkali, such as soap, magnesia, or chalk.

POTASSII ET SODII TARTRAS. Potassium and Sodium Tartrate.

It should contain not less than 99 per cent. of pure Potassium and Sodium Tartrate $[\text{C}_2\text{H}_3(\text{OH})_2(\text{COOK})(\text{COONa}) + 4\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

Colorless, transparent, rhombic prisms, or a white powder, odorless, and having a cooling, saline taste. The crystals slightly effloresce in dry air.

Soluble in about 1.2 parts of water at 25° C. (77° F.) and in less than 1 part of boiling water; almost insoluble in alcohol.

When heated to 74° C. (165.2° F.) the salt fuses to a colorless liquid, which, at a higher temperature, froths, becomes brown, and gradually carbonizes, while inflammable vapors are emitted, having the odor of burning sugar. Finally, a black residue is left.

The aqueous solution of the salt is feebly alkaline to litmus-paper, but it does not effect phenolphthalein.

An aqueous solution of the salt (1 in 10) yields, with an equal volume of acetic acid, a white, crystalline precipitate.

Tests for Identity.—See Potassium, Sodium, and Tartaric Acid in Part V.

Impurities.—Heavy metals and ammonia. Details in Part V.

Assay.—Details in Part V.

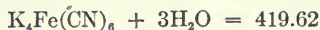
Remarks.—This compound is interesting as one of the two official double salts of tartaric acid, the other being tartar emetic. (See p. 582.) It is made by treating potassium bitartrate with sodium bicarbonate, when the following reaction will occur:



The chemistry of this body can be discussed when the composition of the acid from which it is derived is noticed; hence it is reserved until later (p. 684).

Sodium and potassium tartrate, or *Rochelle salt*, is used as a mild laxative and refrigerant. It is one of the constituents of official Seidlitz powder.

Dose.—8 Gm. (120 grains).

POTASSII FERROCYANIDUM. Potassium Ferrocyanide.

It should contain not less than 99 per cent. of pure Potassium Ferrocyanide and should be kept in well-stoppered bottles.

Large, soft, transparent, yellow, four-sided, monoclinic tabular crystals or prisms; odorless and having a mild, saline taste. Slightly efflorescent on exposure to the air.

Soluble in about 4 parts of water at 25° C. (77° F.) and in 2 parts of boiling water; insoluble in alcohol.

When heated to 60° C. (140° F.) the salt begins to turn white from loss of water of crystallization, and when heated to 100° C. (212° F.) it is rendered anhydrous.

The aqueous solution is neutral to litmus-paper.

Tests for Identity.—See Potassium and Ferrocyanides in Part V.

Impurities.—Carbonate and ferrieyanide. Details in Part V.

Remarks.—Potassium ferrocyanide is interesting as the source of cyanides. It is made from a mixture of nitrogenous animal matter, pearl-ash (potassium carbonate), and scrap-iron, all being heated together in an iron pot.

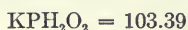
Details of the manufacture of this substance will be studied when we consider the cyanogen preparations. (See p. 745.)

Potassium ferrocyanide is rarely used in medicine. When pure, it is not poisonous, despite the presence of the cyanogen group. A solution of potassium ferrocyanide, we will find later, is used in testing iron (p. 545).

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Under this head mention might be made of the unofficial *potassium ferricyanide*, $K_3Fe(Cn)_6$, which is used in testing for iron in the ferrous form (p. 545). The chemistry of both of these bodies is difficult and will be left to be discussed under the Cyanogen Compounds.

POTASSII HYPOPHOSPHIS. Potassium Hypophosphite.



It should contain not less than 98 per cent. of pure Potassium Hypophosphite [$PO.H_2OK$] and should be kept in well-stoppered bottles. Caution should be observed in dispensing Potassium Hypophosphite, as explosion is liable to occur when it is triturated or heated with nitrates, chlorates, or other oxidizing agents.

White, opaque, hexagonal plates, or crystalline masses, or a granular powder; odorless and having a pungent, saline taste; very deliquescent.

Soluble in about 0.5 part of water and in 7 parts of alcohol at $25^\circ C.$ ($77^\circ F.$); in 0.3 part of boiling water and in 3.6 parts of boiling alcohol; insoluble in ether.

When heated in a dry test-tube, the salt at first loses moisture, and then evolves spontaneously inflammable hydrogen phosphide gas, which burns with a bright yellow flame.

Tests for Identity.—See Potassium and Hypophosphites, Part V.

Impurities.—Carbonates, heavy metals, and arsenic. Details in Part V.

Remarks.—This salt is made by treating calcium hypophosphite with potassium carbonate, as shown in the following equation:

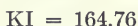


Solution of potassium hypophosphite is removed from the insoluble calcium carbonate by filtration. The solution is then evaporated on a water-bath with great care until a granular mass results. The caution as to evaporation is necessary in heating every hypophosphite, as even at the temperature of the water-bath there is danger of an explosion, due to the formation of the spontaneously combustible phosphine, PH_3 .

Potassium hypophosphite, in common with all hypophosphites, is used largely as a tonic in wasting diseases, and is a constituent of the syrup hypophosphites (U. S. P.).

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

POTASSII IODIDUM. Potassium Iodide.



It should contain not less than 99 per cent. of pure Potassium Iodide and should be kept in well-stoppered bottles.

Colorless, transparent, translucent, or opaque white, cubical crystals, or a white, granular powder, having a peculiar, faint, iodine-like odor, and a pungent, saline, afterward bitter taste. Permanent in dry air and but slightly deliquescent in moist air.

Soluble in 0.7 part of water and in about 12 parts of alcohol at 25° C. (77° F.); in 0.5 part of boiling water and in 6 parts of boiling alcohol; also soluble in 2.5 parts of glycerin.

When heated, the salt decrepitates. At a low red heat it fuses, and at a bright red heat it is volatilized without decomposition.

Its aqueous solution is neutral, or has a slightly alkaline reaction upon litmus-paper.

No residue should be left when 1 Gm. of the salt is dissolved in 2 Cc. of diluted alcohol of specific gravity 0.928 (absence of *less soluble salts*).

Tests for Identity.—See Potassium and Iodides in Part V.

Impurities.—Alkali, iodate, heavy metals, nitrates, nitrites, barium, cyanide, chlorides, bromides, and thiosulphates. Details in Part V.

Assay.—Details in Part V.

The manufacture of this important chemical is similar to that of potassium bromide, only the first process is by treatment of ferric iodide with potassium bicarbonate or carbonate. This is somewhat different from the ferrous bromide reaction in making potassium bromide. As in the bromide process, iron is treated with the halogen element, making the ferrous iodide solution; however, more iodine is added, making the more stable ferric iodide, FeI_3 , and when this is treated with potassium carbonate, the following reaction occurs:



The second process is by treating solution of potassa with iodine, and is closely similar to that for making potassium bromide:



As in the bromide process, the iodate is reduced to iodide by heating with charcoal, viz.:



Potassium iodide occurs in colorless, translucent, cubic crystals, having a faint iodine odor, and a saline, afterward bitter, taste. It is exceedingly soluble in water (1 part to 0.75 at 15° C.), which renders possible the preparation of the saturated solution of potassium iodide, containing one avoirdupois ounce of chemical in a fluidounce of the finished solution.

As mentioned above, the crystals of potassium iodide should be either transparent or very translucent. The larger majority of the commercial iodide is in white, opaque crystals. These are impure, being crystallized from an alkaline medium. It is but fair to the manufacturer, however, to say that the preparation of a neutral potassium iodide is a matter of considerable difficulty, as there is danger of the separation of the iodine turning the crystals brownish.

Potassium iodide is a most valuable alterative.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

POTASSII NITRAS. Potassium Nitrate.



It should contain not less than 99 per cent. of pure Potassium Nitrate $[\text{NO}_3.\text{OK}]$ and should be kept in well-stoppered bottles.

Colorless, transparent, six-sided, rhombic prisms, or a white crystalline powder; odorless and having a cooling, saline, and pungent taste. Slightly hygroscopic in moist air.

Soluble in 3.6 parts of water at 25° C. (77° F.) and in 0.4 part of boiling water; very sparingly soluble in alcohol.

When heated to 353° C. (667.4° F.) the salt melts. At a higher temperature it is decomposed, giving off oxygen at first, and then some of its nitrogen, leaving a residue of potassium nitrate, nitrite, and oxide. When thrown upon red-hot coals the salt deflagrates.

The aqueous solution is neutral to litmus-paper.

Tests for Identity.—See Potassium and Nitrates in Part V.

Impurities.—Heavy metals, iodide, chlorate, and perchlorate. Details in Plate V.

Remarks.—This chemical, commonly called *saltpetre*, has been in popular use for centuries. The original method of manufacture was interesting; being made by the treatment of ashes with decaying nitrogenous matter, such as manure and cattle urine. The mass was allowed to stand under roughly constructed sheds for several months, being occasionally turned so as to present new surfaces to the air. When the fermentation of the mass is completed, it is placed in crude percolators and lixiviated with water, whereby the finished potassium nitrate is separated from the extraneous matter.

Repeated crystallization of this solution eventually results in a pure crystalline nitrate.

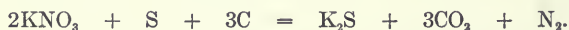
Investigations of the manufacture of saltpetre on these "saltpetre plantations" has shown that the change of the nitrogenous matter to the nitrate is a species of fermentation, being produced by a peculiar microscopic plant called *nitromonas*. This process of saltpetre manufacture was formerly largely used, as it yielded not only all the saltpetre used, but also because this potassium nitrate was the source of nitric acid and all the nitrates.

At present, however, the source of nitric acid is Chile saltpetre (see p. 468), from which potassium nitrate is made, as shown in the following equation:



the resulting salts being separated by fractional crystallization.

One of the largest general uses of potassium nitrate is in the manufacture of *gunpowder*, of which the average formula is potassium nitrate, 75 parts; powdered charcoal, 15 parts; and sulphur, 10 parts, the mass being mixed together, then granulated by wetting the mass, passing it through a sieve, and carefully drying the resulting granules. Gunpowder explodes by the following reaction:



On examination of the above equation it will be seen that the chemical action between the three solids results in the formation of two gases and a comparatively small amount of the solid potassium sulphide. One volume of gunpowder gives 280 volumes of mixed gases, and on this property hinges the whole theory of explosion, which means the sudden evolution of a large volume of gas from a comparatively small quantity of the solid or liquid.

In the potassium sulphide of the above reaction is found an explanation of the peculiar odor of a burned fire-cracker.

In medicine, potassium nitrate is used largely as a diuretic.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Potassium nitrate acts as an oxidizing agent in the explosion of gunpowder, and to this oxidizing action is due the value of potassium nitrate as an antiseptic. Its use as a preservative of meat and other foods is too well known to require extended comment here.

POTASSII PERMANGANAS. Potassium Permanganate.

$$\text{KMnO}_4 = 156.98$$

It should contain not less than 99 per cent. of pure Potassium Permanganate [MnO_3, OK] and should be kept in glass-stoppered bottles, protected from light.

Potassium Permanganate, when in concentrated solution or in the dry condition, should not be brought in contact with organic or other readily oxidizable substances.

Slender, monoclinic prisms, of a dark purple color, almost opaque by transmitted light, and of a blue, metallic lustre by reflected light, odorless, and having a taste at first sweet, but afterward disagreeable and astringent. Permanent in the air.

Soluble in about 15 parts of water at 25°C . (77°F .) and in 3 parts of boiling water; in contact with alcohol it is decomposed.

When heated the salt decrepitates, and at 240°C . (464°F .) it decomposes, yielding oxygen, potassium manganate, and manganese dioxide.

The aqueous solution of the salt is of a deep violet-red color when concentrated and of a rose color when much diluted, and the rose color is discharged by hydrogen sulphide, ferrous sulphate, oxalic acid, alcohol, hydrogen dioxide, and many other readily oxidizable substances, especially if the solution be first rendered acid by sulphuric acid.

An aqueous solution is neutral to litmus-paper.

Tests for Identity.—See Potassium and Permanganates in Part V.

Impurities.—Excess of sulphates, chlorides, nitrates. Details in Part V.

Assay.—Details in Part V.

Remarks.—Potassium permanganate is now given the formula KMnO_4 , in preference to that of the pharmacopœia of 1880, $\text{K}_2\text{Mn}_2\text{O}_8$. It represents the highest known valence of manganese,—the valence VII,—and is an analogue of potassium perchlorate, KClO_4 , which comes from perchloric oxide, Cl_2O_7 , the graphic formula of which was given on p. 365. The permanganate is one of the oxidizing agents, the action of which is explained on p. 425.

It is made by treatment of manganese dioxide with potassium hydroxide and potassium chlorate, this mixture yielding potassium manganate, as shown in the following equation:



It will be observed that potassium manganate (K_2MnO_4) has a formula similar to that of potassium sulphate, in this body the manganese acting with the valence VI. When this green potassium manganate is boiled with water, it is converted into the purple potassium permanganate, as shown in the following equation:



The process occurs in miniature in an important test for manganese, which consists of fusing the three constituents above given on

a piece of platinum foil and then boiling in water. If the fused mass is green and the resulting liquid is pink, it indicates the presence of manganese.

By reason of this change of tint, potassium permanganate has been given the synonym of *mineral chameleon*.

It will be noticed that the products of the final reaction consist of some binoxide of manganese and also potassium hydroxide, and in order to secure the permanganate from this mixture, it is necessary to neutralize the excess of potassium hydroxide with sulphuric acid. The solution of permanganate is then filtered through asbestos to remove the precipitated manganese dioxide, and the filtrate concentrated until crystallization begins.

Potassium permanganate occurs in purple, monoclinic crystals, which possess a blue, metallic luster in reflected light. It is soluble in 16 parts of water and forms a purple-red solution. The pharmacopœia gives a warning that it should never be triturated with easily oxidizable substances, while pills of potassium permanganate should not be made with ordinary excipients, but with petroleum mass (petrolatum and kaolin).

The oxidizing action of potassium permanganate in neutral solution is best shown by the following equation:



This, when reduced into words, means that two molecules of the permanganate yield three molecules of available oxygen. By comparison of this with the oxidizing equation of nitric acid, given on p. 420, it is seen that the process of the two cases is similar.

When permanganate acts in acid solution, two molecules (or one molecule of $\text{K}_2\text{Mn}_2\text{O}_8$ if that is accepted as the formula) liberate five atoms of oxygen, as shown in the following equation:



This is the basis of the use of potassium permanganate in volumetric analysis (p. 1006).

Potassium permanganate is one of the most convenient oxidizing agents for use in organic chemistry. As already stated, nitric acid is the oxidizing agent generally used in the ordinary processes of oxidation we meet in pharmacy. The acid, however, acts too violently for use in many organic oxidations, and for such purposes either potassium permanganate or chromic acid is employed. The only pharmaceutical application of this use of permanganate is in purifying alcoholic distillates. In such cases it is well to note that the permanganate decomposes with the formation of not only insoluble manganese dioxide, but also potassium hydroxide, which will go into solution. Therefore, before such purified alcohol can be employed, it should be redistilled.

Potassium permanganate is a valuable antiseptic in medicine, and is largely used in surgical practice. One advantage it possesses over the ordinary oxidizing antiseptics is its comparative freedom from caustic action. In practice it is frequently prescribed in the form of *Condy's fluid*, which consists of 128 grains permanganate to one pint of distilled water.

Dose.—65 milligrammes (1 grain), preferably in a fine powder in capsules.

POTASSII SULPHAS. Potassium Sulphate.

It should contain not less than 99 per cent. of pure Potassium Sulphate $[\text{SO}_2(\text{OK})_2]$.

Hard, colorless, transparent, six-sided, rhombic prisms terminated by pyramids, or a white powder, odorless, and having a somewhat bitter, saline taste. Permanent in the air.

Soluble in about 9 parts of water at 25°C . (77°F .) and in 4 parts of boiling water; insoluble in alcohol.

When heated the crystals decrepitate. At a bright red heat they fuse, and at a white heat the salt suffers partial decomposition.

The aqueous solution is neutral to litmus-paper.

Tests for Identity.—See Potassium and Sulphates in Part V.

Impurities.—Heavy metals and arsenic. Details in Part V.

Remarks.—This body occurs in nature in the ore, *kainite*, of the composition $\text{K}_2\text{SO}_4\text{MgSO}_4\text{MgCl}_2\cdot 5\text{H}_2\text{O}$. It is made artificially by treating either potassium nitrate or potassium chloride with sulphuric acid. It is in the form of colorless, transparent, rhombic crystals, soluble in $9\frac{1}{2}$ parts of water. It is interesting to note that these crystals contain no water of crystallization, thereby differing from the otherwise similar sodium sulphate, which contains ten molecules of water of crystallization.

Potassium sulphate is employed as a diuretic and refrigerant. Its use is rather limited, especially as the pharmacopœia has omitted its use in the manufacture of Dover's powder, the original recipe for this pharmaceutic directing that the ipecac and opium be triturated with crystallized potassium sulphate; but in the present pharmacopœia sugar of milk is substituted for this salt. (See p. 304.)

Dose.—2 Gm. (30 grains).

CHAPTER XXVI**SODIUM.**

Symbol, Na. Atomic weight, approximately 23.

THE symbol Na is an abbreviation of the words *natrîum*, this being derived from natron, which was from the Greek word *nitron*, which was the original name of the carbonate. Our English word sodium is derived from soda, which comes from the word *sodash*, this hinting its preparation from the ashes of "sods," or marine plants.

Sodium, like potassium, was isolated by Sir Humphrey Davy in 1807 by the electrolysis of the hydrate.

Occurrence.—The most abundant source of sodium is common salt, or sodium chloride, this being a constituent of sea-water, but chiefly obtained in commerce either from mines or by the evaporation of brine from salt-wells. Sea-water is a scarcely available source of sodium chloride, because therein are found chlorides of many other elements which are separated with difficulty.

Manufacture.—The element sodium is isolated by a process quite similar to that by which we obtain potassium, viz., by heating carbonate with coal, chalk being added as a flux. This process is not so dangerous as is the preparation of potassium, inasmuch as the sodium carbonyl, NaCO , does not solidify in the delivery tube. (See p. 431.)

Metallic sodium is now being made by treating sodium hydrate with iron carbide at a temperature of 800°C .

It is also obtained by the electrolysis of sodium hydrate in the same manner employed in isolating potassium.

Properties.—Sodium is a soft, silvery-white metal, resembling potassium, and, like it, is so easily oxidized that it must be kept under petroleum. When metallic sodium and mercury are rubbed together, an amalgam results which, when brought into contact with moisture, ignites. This mixture has been used as a cigar-lighter, but its danger was such that it never became popular.

The characteristic tests of the element and its salts are the yellow color that it communicates to a colorless flame; a white precipitate formed when a solution of sodium salt is brought into contact with a solution of pyroantimoniate of potassium.

The sodium flame is counted among the most delicate of tests, it having been estimated that $\frac{1}{65,000,000}$ grain will respond thereto. By reason of its sensitiveness the positive testing of sodium with the flame is difficult, as almost anything tested will give a yellow flame by reason of the presence of minute traces of sodium. Hence the chemist trains his eye to a rough estimation of the quantity shown by the intensity of the flame, rather than by the bare fact of the presence of a yellow color.

The following compounds of sodium and their preparations are official:

Sodium hydroxide.....	90 per cent. absolute.	NaOH .
Solution of sodium-hydroxide.....	5 " "	NaOH .
Sodium acetate.....	99.5 " "	$\text{NaC}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$.
Sodium benzoate.....	99 " "	$\text{NaC}_7\text{H}_5\text{O}_2$.
Sodium bicarbonate.....	99 " "	NaHCO_3 .
Lozenges of sodium bicarbonate.....	(pharmaceutic).	
Sodium bisulphite.....	90 per cent. absolute.	NaHSO_3 .
Sodium borate.....	99 " "	$\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$.
Sodium bromide.....	97 " "	NaBr .
Monohydrated sodium carbonate.....	99 " "	$\text{Na}_2\text{CO}_3 + \text{H}_2\text{O}$.
Sodium chlorate.....	99 " "	NaClO_3 .
Sodium chloride.....	99 " "	NaCl .
Sodium citrate.....	97 " "	$2\text{Na}_2\text{C}_6\text{H}_7\text{O}_7 + 11\text{H}_2\text{O}$.
Sodium hypophosphite.....	98 " "	$\text{NaPH}_2\text{O}_2 + \text{H}_2\text{O}$.
Sodium iodide.....	98 " "	NaI .
Sodium nitrate.....	99 " "	NaNO_3 .
Sodium nitrite.....	90 " "	NaNO_2 .
Sodium phenolsulphonate.....	99 " "	$\text{NaC}_6\text{H}_4\text{SO}_3\text{S} + 2\text{H}_2\text{O}$.
Sodium phosphate.....	99 " "	$\text{Na}_2\text{HPO}_4 + 12\text{H}_2\text{O}$.
Exsiccated sodium phosphate.....	99 " "	Na_2HPO_4 .
Effervescent sodium phosphate.....	20 per cent. exsiccated sodium phosphate.	
Compound solution of sodium phosphate	contains 1000 Gm. sodium phosphate to the liter.	
Sodium pyrophosphate.....	99 per cent. absolute.	$\text{Na}_4\text{P}_2\text{O}_7 + 10\text{H}_2\text{O}$.
Sodium salicylate.....	99.5 " "	$\text{NaC}_7\text{H}_5\text{O}_3$.
Sodium sulphate.....	99 " "	$\text{Na}_2\text{SO}_4 + 10\text{H}_2\text{O}$.
Sodium sulphite.....	96 " "	$\text{Na}_2\text{SO}_3 + 7\text{H}_2\text{O}$.
Sodium thiosulphate.....	98 " "	$\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$.

We might also add:

Potassium and sodium tartrate (considered among the potassium salts).
 Solution of chlorinated soda (considered among the chlorine preparations).
 Sodium arsenate (considered among the arsenic compounds).
 Exsiccated sodium arsenate (considered among the arsenic compounds).
 Solution of sodium arsenate (considered among the arsenic compounds).

SODII HYDROXIDUM. Sodium Hydroxide.

NaOH = 39.76

[SODA, PHARM. 1890]

It should contain not less than 90 per cent. of pure anhydrous Sodium Hydroxide and not more than 2 per cent. of other inorganic substances, with the exception of water. It should be kept in well-stoppered bottles made of hard glass.

Dry, white flakes, fused masses, or translucent or opaque white pencils, showing a crystalline fracture, odorless, and having a caustic taste. *Great caution is necessary in tasting and handling it*, as it rapidly destroys organic tissues. Exposed to the air, it rapidly deliquesces, absorbs carbon dioxide, and becomes covered with a dry coating of carbonate.

Soluble in about 1 part of water at 25° C. (77° F.) and in 0.8 part of boiling water; very soluble in alcohol.

When heated to about 525° C. (977° F.) Sodium Hydroxide melts to a clear, oily liquid, and at a bright red heat it is slowly volatilized unchanged. When introduced into a non-luminous flame, it imparts to it an intense yellow color.

A solution of Sodium Hydroxide, even when greatly diluted, gives an alkaline reaction with red litmus-paper.

The aqueous solution (1 in 20) should be perfectly clear and colorless (absence of organic matter and insoluble impurities).

Tests for Identity.—Given above.

Impurities.—Organic matter (see above); potassium, heavy metals, excess of carbonate, excess of silicate. Details in Part V.

Assay.—Details in Part V.

Remarks.—Soda is prepared by the evaporation of a solution of soda. It occurs in irregular lumps or in cylindric sticks, just as does potassa, and is, in fact, very similar to potassa, being decidedly caustic and possessing the same soapy taste, which is a distinguishing characteristic of the alkalis. It differs from potassa, however, in being less deliquescent.

LIQUOR SODII HYDROXIDI. Solution of Sodium Hydroxide.

[LIQUOR SODÆ, PHARM. 1890]

An aqueous solution, containing about 5 per cent. of Sodium Hydroxide [NaOH=39.76].

<i>Recipe.</i> —Sodium Hydroxide, fifty-six grammes.....	56 Gm.
Distilled Water, nine hundred and forty-four grammes.....	944 Gm.
To make one thousand grammes..	1000 Gm.

Dissolve the Sodium Hydroxide in the Distilled Water.

The Sodium Hydroxide used in this process should be of the full strength and quality directed by the Pharmacopœia (90 per cent.). Sodium Hydroxide of any other strength, however, may be used, if a proportionately larger or smaller quantity be taken; the proper amount for the above formula being ascertained by dividing 5040 by the percentage of absolute Sodium Hydroxide contained therein.

Solution of Sodium Hydroxide should be kept in bottles made of green glass, and provided with glass stoppers coated with paraffin or petrolatum.

A clear, colorless liquid, odorless, having a very acrid and caustic taste, and a strongly alkaline reaction.

Specific gravity: about 1.056 at 25° C. (77° F.).

It should conform to the reactions and tests for an aqueous solution of sodium hydroxide given under *Sodii Hydroxidum*.

Assay.—Details in Part V.

Remarks.—Solution of soda is an aqueous solution, containing 5 per cent. of absolute NaOH. It is prepared by a process analogous to that of the manufacture of the solution of potassa, by treatment of sodium carbonate with calcium hydrate, as shown in the following equation:



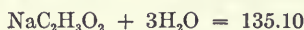
Note that the words sodium and soda are not to be used indiscriminately. Sodium is an element, while soda is the hydrate of that element. Note the absurdity and real danger of the term “soda-water,” which is a solution of carbonic oxide in water.

The solution of soda is used as an antacid.

Dose.—1 Cc. (15 minims), very largely diluted.

Antidotes.—(See p. 435.)

SODII ACETAS. Sodium Acetate.



It should contain in an uneffloresced condition not less than 99.5 per cent. of pure Sodium Acetate [$\text{CH}_3\text{COONa} + 3\text{H}_2\text{O}$] and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms, or a granular, crystalline powder; odorless and having a cooling, saline taste. Efflorescent in warm, dry air.

Soluble in about 1 part of water and in 23 parts of alcohol at 25° C. (77° F.); and in all proportions of boiling water and of boiling alcohol.

When heated to 60° C. (140° F.) the salt begins to liquefy. At 123° C. (253.4° F.) it becomes dry and anhydrous; at 315° C. (599° F.) it is decomposed, with evolution of inflammable, empyreumatic vapors, leaving a black residue of sodium carbonate and carbon, which imparts to a non-luminous flame an intense yellow color, gives an alkaline reaction with litmus-paper, and effervesces with acids.

An aqueous solution of the salt (1 in 20) should be alkaline to red litmus-paper, but should not affect phenolphthalein.

Tests for Identity.—See Sodium and Acetates in Part V.

Impurities.—Excess of potassium, arsenic, and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—*Sodium acetate*, like the potassium salt, is made from acetic acid, and the “stock” salt of the metal. It will be remembered that the “stock” salt of potassium—the one usually employed in the manufacture of ordinary salts of that metal—was the bicarbonate, the carbonate being inadmissible by reason of its indefinite quality. With sodium compounds the “stock” salt is the carbonate, as it is readily crystalline and quite stable if kept in a well-corked bottle. Its tendency is toward efflorescence—not deliquescence—such is the usual inclination of sodium compounds; just as soda soap—Castile soap—grows hard and dry on standing: while potassa soap—soft soap—and other potassium compounds are usually deliquescent.

Sodium acetate is made by the action of acetic acid on sodium

carbonate, and the latter salt is used in the manufacture of most sodium compounds. Let us compile a list of official sodium salts prepared from the carbonate:

Sodium benzoate from benzoic acid and sodium carbonate.
 Sodium bisulphite from sulphurous acid and sodium carbonate.
 Sodium bromide from ferrous bromide and sodium carbonate.
 Sodium hypophosphite from calcium hypophosphite and sodium carbonate.
 Sodium iodide from ferrous iodide and sodium carbonate.
 Sodium phosphate from acid calcium phosphate and sodium carbonate.
 Sodium salicylate from salicylic acid and sodium carbonate.
 Sodium sulphite from sulphurous acid and sodium carbonate.
 Sodium sulphocarbolate from barium sulphocarbolate and sodium carbonate.
 Solution of sodium silicate (U. S. P. 1890) from sand and sodium carbonate.
 Sodium arsenate from arsenous acid, sodium nitrate, and sodium carbonate.

The pharmaceutic value of sodium carbonate can thus be seen.

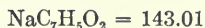
Sodium acetate occurs in the form of colorless, transparent crystals, or in a granular powder, and must be kept in well-stoppered bottles by reason of its deliquescence. It is, however, by no means so deliquescent as is the corresponding potassium compound.

Dose.—1 Gm. (15 grains).

SODII ARSENAS. Sodium Arsenate.

This will be considered among the arsenic compounds (p. 577).

SODII BENZOAS. Sodium Benzoate.



It should contain not less than 99 per cent. of pure Sodium Benzoate [$\text{C}_6\text{H}_5\text{COONa}$] and should be kept in well-stoppered bottles.

A white, amorphous, granular or crystalline powder; odorless and having a sweetish astringent taste. Permanent in the air.

Soluble in 1.6 parts of water and in 43 parts of alcohol at 25°C . (77°F .); in 1.3 parts of boiling water and in 12 parts of boiling alcohol.

When heated the salt melts, emits vapors having the odor of benzoic acid, then chars, and finally leaves a residue of sodium carbonate and carbon. To a non-luminous flame it imparts an intense yellow color.

Its aqueous solution is neutral or slightly alkaline to litmus-paper.

Tests for Identity.—See Sodium and Benzoates in Part V.

Impurities.—Heavy metals. Details in Part V.

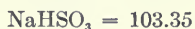
Assay.—Details in Part V.

Remarks.—Of this rather uninteresting salt, little is necessary to be stated beyond the pharmacopœial description. Its method of manufacture has been outlined above.

Dose.—1 Gm. (15 grains).

Sodium bicarbonate, NaHCO_3 , will be considered along with sodium carbonate, as the two are so mutually dependent that they should be studied together.

SODII BISULPHIS. Sodium Bisulphite.



It should contain not less than 90 per cent. of pure Sodium Bisulphite and should be kept in a cool place, in small, completely filled, well-stoppered bottles.

Opaque, prismatic crystals, or a granular powder, exhaling an odor of sulphur dioxide, and having a disagreeable, sulphurous taste. Exposed to the air the salt loses sulphur dioxide, and is gradually oxidized to sulphate.

Soluble in 3.5 parts of water and in 70 parts of alcohol at 25° C. (77° F.); in about 2 parts of boiling water and in 49 parts of boiling alcohol.

When strongly heated the salt decrepitates, emits vapors of sulphur and of sulphur dioxide, and leaves a residue of sodium sulphate. To a non-luminous flame it imparts an intense yellow color.

Its aqueous solution gives an acid reaction with blue litmus-paper.

Tests for Identity.—See Sodium and Sulphites in Part V.

Impurities.—Thiosulphates, heavy metals. Details in Part V.

Assay.—Details in Part V.

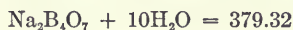
Sodium bisulphite is another of the acid salts described on p. 378. Being a bisulphite, it is a derivative of sulphurous acid, which itself is derived from sulphurous oxide. This oxide shows sulphur with the valence iv, and its formula is SO_2 , as is shown graphically on p. 365. $\text{SO}_2 + \text{H}_2\text{O} = \text{H}_2\text{SO}_3$, which is sulphurous acid. When all the hydrogen of sulphurous acid is replaced by sodium, we get sodium sulphite, Na_2SO_3 ; but when only one of the hydrogen atoms is replaced—by one atom of sodium—we get the bisulphite, NaHSO_3 .

This salt is made by passing sulphurous acid gas through solution of sodium carbonate to saturation. The solution is then cooled, when the salt crystallizes out.

Sodium bisulphite is an unstable solid, rapidly losing sulphur dioxide and becoming oxidized to sulphate. For this reason the pharmacopœia directs that it be kept in a cool place, in small, well-stoppered bottles, which are to be completely filled. Sodium bisulphite is used as a reducing agent in organic chemistry, and is particularly valuable in the bleaching industry for the removing of the last traces of chlorine from the bleached fabric. Substances used for this purpose are known as *antichlors*. It is comparatively little used in medicine, but when so used, it is as an antiseptic.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

SODII BORAS. Sodium Borate.



It should contain in the uneffloresced condition not less than 99 per cent. of pure sodium tetraborate and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms, or a white powder, inodorous, and having a sweetish, alkaline taste. Slightly efflorescent in warm, dry air.

Soluble in 20.4 parts of water at 25° C. (77° F.) and in 0.5 part of boiling water; insoluble in alcohol; it is soluble in 1 part of glycerin at 80° C. (176° F.).

When heated, the salt at first loses part of its water of crystallization, then melts, and, when further heated, swells up and forms a white, porous mass.

At a red heat it loses all of its water of crystallization (47 per cent.) and fuses to a colorless glass. To a non-luminous flame it imparts an intense yellow color.

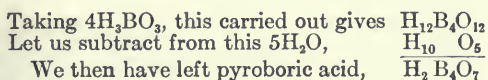
An aqueous solution (1 in 20) colors red litmus-paper blue and yellow turmeric paper reddish-brown. After being acidulated with hydrochloric acid, the solution colors blue litmus-paper red; yellow turmeric paper remains unchanged at first, but on drying becomes brownish-red, and this color is temporarily changed to bluish-black by moistening with ammonia water.

Tests for Identity.—See Sodium and Borates in Part V.

Impurities.—Carbonates, bicarbonates, heavy metals, phosphate, nitrates. Details in Part V.

Sodium borate—ordinary *borax*—is a valuable and interesting chemical. Its formula, $\text{Na}_2\text{B}_4\text{O}_7$, seems most difficult, and contradictory to the formula of boric acid already given— H_3BO_3 , but investigation clears up the matter. Remember that the valence of boron is III; that boric acid is a true ortho-acid— $\text{B}(\text{OH})_3$ or H_3BO_3 . True sodium borate has the formula Na_3BO_3 , and what we call sodium borate is not the true salt, but a "*pyroborate*." The Greek word for fire is "*pyros*," and all chemicals bearing the prefix "*pyro*" are bodies formed under influence of high heat. In passing, such examples as "*pyroligneous acid*" (crude acetic acid), "*pyrogallic acid*," and "*pyrophosphoric acid*" may be cited. Like these, pyroboric acid and pyroborates are made by subjecting the mother substance to heat.

Let us see what will occur when boric acid is heated. It will, of course, like all ortho-acids, lose water, and, under proper conditions, in a most peculiar ratio:



the sodium salt of which, $\text{Na}_2\text{B}_4\text{O}_7$, is official borax, which is a natural product, being found in certain lakes of Thibet, Tartary, and India. The impure borax, obtained by spontaneous evaporation of the waters of these lakes, is called *tincal*. At the present time practically all the American borax is obtained from the borax lakes found in the Death Valley of Nevada. It is also made by treating the native boric acid of Tuscany with sodium carbonate.

Borax occurs either in colorless, transparent prisms or in a white powder, odorless, and having a sweetish, alkaline taste.

It is soluble in 16 parts of water and in 1 part of glycerin. When heated, it fuses and solidifies into a transparent solid called borax glass. This borax glass is used quite largely in analytic work under the name of borax bead, the chemical being fused on a loop of platinum wire, thereby forming a minute globule of borax glass. When this body is melted with certain metallic salts, the body is given characteristic colors. (See p. 985.)

Borax is one of the most valuable of the sodium compounds. It is used in numerous operations where a mild alkali is indicated, it completely possessing the character of an alkali, and yet acts in a manner much milder than does either sodium carbonate or the bicarbonate. This explains its extensive use in cleansing delicate fabrics, such as fine cloth or hair, where stronger alkalis would act disastrously. It is also a valuable antiseptic, and for such purposes it is used as a preservative for meats and food. In medicine it is used as a mild antiseptic, chiefly externally.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Compound Solution of Sodium Borate (N. F.), or *Dobell's Solution*, is rather largely used as a nasal douche.

The recipe calls for 15 Gm. sodium borate, 15 Gm. sodium bicarbonate, 3 Gm. phenol, 35 Cc. glycerin, and water to make 1000 Cc. For details see the National Formulary.

SODII BROMIDUM. Sodium Bromide.

It should contain, when dried, not less than 97 per cent. of pure Sodium Bromide and should be kept in well-stoppered bottles.

Colorless or white cubical crystals, or a white granular powder, odorless, and having a saline, slightly bitter taste. The salt absorbs moisture from the air without deliquescent.

Soluble in about 1.7 parts of water and in 12.5 parts of alcohol at 25° C. (77° F.); in 0.8 part of boiling water and in 11 parts of boiling alcohol.

When heated to a bright red heat the salt melts, and, at a somewhat higher temperature, slowly volatilizes without decomposition. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution (1 in 20) is neutral or shows a faintly alkaline reaction with red litmus-paper.

Tests for Identity.—See Sodium and Bromides in Part V.

Impurities.—Excess of alkali, iodides, heavy metals, bromates, and barium. Details in Part V.

Assay.—Details in Part V.

Remarks.—This chemical is made by methods analogous to that employed in making potassium bromide; that is, by treatment of sodium hydrate with bromine, and the subsequent heating of the mixture with charcoal, or by the double decomposition of ferrous bromide with sodium bicarbonate.

Sodium bromide occurs in white, cubic crystals which are soluble in 1.2 parts of water. The crystals attract moisture, but do not liquefy therein. It is used as a mild sedative, closely resembling potassium bromide in action.

Dose.—1 Gm. (15 grains).

SODII CARBONAS MONOHYDRAS. Monohydrated Sodium Carbonate.

It should contain not less than 85 per cent. of pure anhydrous Sodium Carbonate $[\text{CO}(\text{ONa})_2]$, corresponding to not less than 99.5 per cent. of the crystallized monohydrated salt.

A white, crystalline, granular powder, odorless, and having a strongly alkaline taste. When exposed to the air, under ordinary conditions, it absorbs only a slight percentage of moisture; exposed to warm, dry air at or above 50° C. (122° F.) the salt effloresces, and at 100° C. (212° F.) it loses its water of crystallization (14.52 per cent.).

Soluble in 2.9 parts of water at 25° C. (77° F.) and in 1.8 parts of boiling water; insoluble in alcohol and in ether; soluble in 8 parts of glycerin.

The aqueous solution shows an alkaline reaction with red litmus-paper, and effervesces strongly with acids. To a non-luminous flame the solution imparts an intense yellow color.

Tests for Identity.—See Sodium and Carbonates in Part V.

Impurities.—Heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—Sodium carbonate, the next chemical demanding our attention, is, with the sole exception of sulphuric acid, the most widely used manufactured product in the chemical world. To mention all the many important industries in which sodium carbonate is a potent factor would occupy too much space, but chief among the branches of

trade to which sodium carbonate is essential are soap-making and glass manufacture.

In early time the only source of sodium carbonate was by evaporation of water from certain soda lakes, such as *trona* from Egypt, or from the crude salt obtained by lixiviating the ashes of sea-weeds, rich in soda, such as *barilla* from Spain. The output from these sources was rather limited, and as the soap and glass industries became more important, the need for cheap sodium carbonate became more and more imperative, and, accordingly, a prize was offered by the Paris Academy, in 1782, for the best method of artificial production of this valuable alkali, and the prize was awarded to Nicholas Leblanc. In 1794 the French government took charge of his factory.

A most striking proof of the value of the process is shown by the fact that to this day a large amount of sodium carbonate is still manufactured by the Leblanc process.

In passing, it should be mentioned that Leblanc's process, like most other great discoveries, did not originate solely with the man whose name it bears. Duhamel in 1736 converted sodium chloride successfully into sodium sulphate, sodium sulphide, sodium acetate, and lastly into sodium carbonate. Marggraf, 1759, shortened the transformation by converting sodium chloride into sodium sulphate, then into sodium nitrate, and this, by strong heat, into the oxide which, on standing, absorbed enough carbon dioxide from the air to change into carbonate.

It is even said that the last portion of the Leblanc process (use of coal and limestone) was suggested to him by De la Mitherie, but be this as it may, it was Leblanc established the first factory for the manufacture of sodium carbonate, and to him the real credit is due, although it has been recently claimed that even this credit belongs to another, the chemist Dize.

Leblanc chose as the sodium compound on which to begin operations that which was most widely distributed; that body which was found both on land and in the sea—sodium chloride or common salt. This is the cheapest sodium compound, and the only reason it is not used as the "stock salt" in chemical manufacturing—in making other sodium salts—is because of the difficulty with which it decomposes, there being very few acids which can break it up. Among these is sulphuric acid, the reaction being $2\text{NaCl} + \text{H}_2\text{SO}_4 = \text{Na}_2\text{SO}_4 + 2\text{HCl}$. Note that hydrochloric acid, HCl , is a side product in the process. In England, when the manufacture was first begun, the acid was allowed to escape with the air through tall chimneys, with most terrible results—as the vapor mingled with the aqueous vapor and fell with the latter as rain, destroying vegetation and ruining health. So great was the damage that stringent laws were enacted prohibiting the discharge of the acid vapor. Thereupon the manufacturers took to condensing the vapors and disposing of the hydrochloric acid, and by curious change of times, at present, the keen competition has so lessened the price of sodium carbonate that the manufacturers gain much of their profit from the sale of the acid.

To return to the process, which we have only followed to the formation of sodium sulphate, and which seems as far off as was the chloride. But Leblanc's genius solved the problem of conversion. He realized that while the *sulphate* was a very stable body, the *sulphide* was quite weak—could be replaced even by the CO_3 group.

The changing of the sulphate, Na_2SO_4 , to the sulphide, Na_2S , is

very simple. It is merely a process of reduction, and is performed by the reducing agent we used in the manufacture of sulphurous acid—the element carbon, usually in the form of common coal. The reaction is as follows:



Having converted the strong sulphate into the easily dissociated sulphide, the next step is to bring about a double decomposition between this sulphide and some cheap carbonate. The carbonate best answering this requirement is the calcium salt, which is abundantly found as marble, limestone, chalk, and oyster shells. This salt is accordingly employed, and the reaction is:

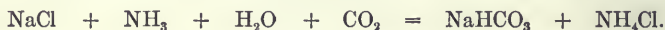


In practice, the sulphate is heated with coal and limestone at the same time, hence the two actions run simultaneously.

The next process we have to consider is that which leads to the sodium bicarbonate instead of to the carbonate. This is the *Solvay process*, and is one of the most clear-cut processes we meet in chemistry, as both reaction products are valuable, and there is little waste-product. It is also called the “ammonia soda process,” after the constituents employed.

Like Leblanc’s process, it starts off with that most abundant source of sodium, the chloride.

A solution of salt is placed in a tin-lined vat and is saturated with ammonia gas. This liquid, on cooling, is pumped into a second vat, where it absorbs carbon dioxide gas and forms sodium bicarbonate and ammonium chloride by the following reaction:



The process is comparatively modern, and is likely eventually to replace the Leblanc process, as it is less wasteful and yields a finer and more expensive product.

The third method for obtaining the carbonic acid compounds of sodium is by the *cryolite process*, which is named from the Greenland mineral, which is the stock salt. Cryolite is a fluoride of aluminum and sodium, the formula of which is not so difficult if one only remembers that fluorine forms compounds entirely analogous to the chlorides, the bromides, and the iodides. Aluminum fluoride is AlF_3 , since aluminum is trivalent and the linkage is graphically as follows:



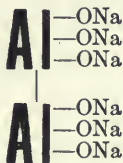
When written double, the formula is Al_2F_6 . Sodium fluoride is NaFl —exactly analogous to NaCl . The proportions in which these two fluorides combine are six molecules of NaFl to one of Al_2F_6 .

The method of manufacture of the carbon from cryolite has not the extended use that Leblanc’s and Solvay’s processes enjoy, it being practically a monopoly of one corporation.

Cryolite is first heated with limestone, and is thus converted into sodium aluminate and calcium fluoride, as shown in the following equation:



The graphic formula of sodium aluminate is supposed to be as follows:



That is, aluminum hydrate (p. 534) with its six atoms of hydrogen replaced by six atoms of sodium.

It is met with in analytic chemistry, one of the tests for aluminum being that any soluble salt of this metal, when treated with sodium hydroxide, yields a gelatinous precipitate, aluminum hydroxide, which is soluble in an excess of sodium hydroxide (p. 534).

In other words, when an excess of sodium hydroxide is added, the aluminum hydroxide combines with it to form a soluble aluminate of sodium, just described. This reaction is also employed in the separation of iron from aluminum in analytic work.

Returning to our cryolite process: the aluminate of sodium is converted into sodium carbonate by treatment with carbon dioxide gas by the following reaction:



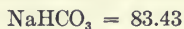
The commercial value of this method of manufacturing sodium carbonate is due not only to the output of sodium carbonate, but also to the fact that the side-products—aluminum oxide and calcium fluoride—are worked up into the alums and the fluorine derivatives respectively.

Sodium carbonate is an antacid, but is not used much internally, the less irritating bicarbonate being preferred.

Dose.—250 milligrammes (4 grains).

Sodii Carbonas (U. S. P. 1890) was the crystalline decahydrated form; that is, containing ten molecules of water of crystallization— $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$. This is what is familiarly called *washing-soda*, and had the disadvantage of being very efflorescent; so much so that the writer has seen crystals of the salt lose water while still in the crystallizing vat. This made it a matter of difficulty to employ the salt in work involving molecular proportions; hence in the last revision the monohydrated salt was given official recognition. Because the monohydrated salt is both stable and concentrated, it replaced both the crystalline and the exsiccated carbonate of the old pharmacopœia.

Sodii Carbonas Exsiccatus (U. S. P. 1890).—This substance is made by taking decahydrated sodium carbonate and exsiccating it at a temperature of about 45°C . (115°F .) until the product has lost half its weight. The object of using the heat of a water-bath is in order to avoid the possible loss of carbon dioxide. The dry carbonate is used for the same purpose as in the decahydrated, but is about twice the strength, and is preferable for filling prescriptions of pills and powders.

SODII BICARBONAS. Sodium Bicarbonate.

It should contain not less than 99 per cent. of pure Sodium Bicarbonate [$\text{CO}(\text{OH})\text{-(ONa)}$] and should be kept in well-closed vessels, in a cool place.

A white, opaque powder, odorless, and having a cooling, mildly alkaline taste. Permanent in dry, but slowly decomposed in moist air.

Soluble in 12 parts of water at 15°C . (59°F .); above this temperature the solution gradually loses carbon dioxide, and at boiling heat the salt is entirely converted into normal carbonate; insoluble in alcohol.

When heated, the salt is decomposed into normal carbonate, water, and carbon dioxide, and, finally, at 100°C . (212°F .), loses about 36.9 per cent. of its weight. At a bright red heat it melts. To a non-luminous flame it imparts an intense yellow color.

The solution, when freshly prepared with cold distilled water, without shaking, gives a slightly alkaline reaction with litmus-paper. The alkalinity increases by standing, agitation, or increase of temperature.

With acids the solution effervesces strongly.

If Sodium Bicarbonate be heated in a test-tube, no odor of ammonia should be evolved.

If 1 Gm. of the salt be dissolved in 19 Cc. of water, it should yield a perfectly clear and colorless solution, leaving no residue.

Tests for Identity.—See Sodium and Carbonates in Part V.

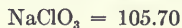
Impurities.—Ammonia (see above), excess of carbonate, sulphocyanate, and heavy metals. Details in Part V.

Assay.—Details in Part V.

The process for making this salt is the Solvay process just described. It can also be made by treating sodium carbonate with carbon dioxide, but such a process is no longer much employed.

Sodium bicarbonate usually occurs in the form of a white, opaque powder, which is soluble in 11.3 parts of water; therefore much less soluble than is the carbonate. Sodium bicarbonate is a popular antacid, being comparatively free from irritating action.

Dose.—1 Gm. (15 grains).

SODII CHLORAS. Sodium Chlorate.

It should contain not less than 99 per cent. of pure Sodium Chlorate [ClO_2ONa].

This salt should be kept in well-stoppered bottles, and *great caution should be observed* in handling it, as dangerous explosions are liable to occur when it is heated, or subjected to concussion or trituration with organic substances (cork, tannic acid, sugar, etc.), or with sulphur, antimony sulphide, phosphorus, or other easily oxidizable substances.

Colorless, transparent crystals (principally cubes with tetrahedral facets), or a crystalline powder; odorless and having a cooling, saline taste. Permanent in dry air.

Soluble in about 1 part of water and in about 100 parts of alcohol at 25°C . (77°F .); in 0.5 part of boiling water and in about 40 parts of boiling alcohol; also soluble in about 5 parts of glycerin.

When heated the salt melts, then gives off oxygen (about 45 per cent. of its weight), and finally leaves a residue of sodium chloride, readily soluble in water, and yielding, with a silver nitrate T.S., a white, curdy precipitate, insoluble in nitric acid. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution of Sodium Chlorate (1 in 20) is neutral to litmus-paper.

Tests for Identity.—See Sodium and Chlorates in Part V.

Impurities.—Potassium, lead, and copper. Details in Part V.

Remarks.—This chemical is made like potassium chlorate, by treating calcium chlorate with sodium chloride.

It can also be made by treating potassium chlorate with sodium bitartrate by the following equation:



The potassium bitartrate produced in the reaction precipitates from the solution as it does in the Fothergill process (p. 414).

Sodium chlorate so closely resembles potassium chlorate that extended comment is unnecessary. Like potassium chlorate, it must be handled carefully in reference to trituration with oxidizable bodies, in order to avoid an explosion.

Its only advantage over potassium chlorate is that it is more soluble in water, 1 part of the sodium salt dissolving in 1.1 parts of water, while 1 part of potassium chlorate requires 16.5 parts of water to make a solution. Its uses are about the same as those of potassium chlorate.

Dose.—250 milligrammes (4 grains).

SODII CHLORIDUM. Sodium Chloride.

$\text{NaCl} = 58.06$

It should contain when dried not less than 99 per cent. of pure Sodium Chloride.

Colorless, transparent, cubical crystals, or a white, crystalline powder, odorless, and having a purely saline taste. Permanent in dry air.

Soluble in 2.8 parts of water at 25° C. (77° F.) and in 2.5 parts of boiling water; almost insoluble in alcohol.

When heated, the salt decrepitates. At a red heat it fuses, and at a white heat it is slowly volatilized and partly decomposed. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution of the salt is neutral to litmus-paper.

Tests for Identity.—See Sodium and Chlorides in Part V.

Impurities.—Heavy metals, bromide, and iodide. Details in Part V.

Assay.—Details in Part V.

Sodium chloride, or *common salt*, is one of the most valuable and ancient of the chemicals. It is essential to animal economy, hence it has been used from the earliest days. As mentioned above, it is the chief source of metallic sodium and of the sodium compounds, and is found in the native state widely distributed.

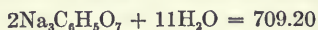
Its presence in sea-water, salt-wells, and salt mines has been mentioned, so here it is only necessary to state that, among the chief deposits of salt, are the mines near Cracow, in eastern Poland; near Salzburg, in Tyrol; and the Petit Anse mines of southwest Louisiana. In many parts of the world are found deposits of salt of not sufficient area to justify mining. In these cases shafts are sunk until the deposit is reached, water poured in, and when saturated with the salt, is pumped out and the solution evaporated. Such process is now carried on in Cleveland, O. Another source of salt is the natural salt springs found in certain sections of the world. There are many of these in Germany of insufficient concentration to justify the use of heat for complete evaporation, and in such cases the major portion

of the water is removed by the application of spontaneous evaporation, explained on p. 89.

Normal saline solution, which is in some demand, is a $\frac{9}{10}$ of 1 per cent. solution of sodium chloride. It is presumably of the same saline strength as blood-serum, and is used for transfusion operations.

Dose.—As emetic, 16 Gm. (240 grains).

SODII CITRAS. Sodium Citrate.



It should contain not less than 97 per cent. of pure Sodium Citrate $[\text{2C}_3\text{H}_4(\text{OH})(\text{COONa})_3 + 11\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

A white, granular powder, odorless, and having a cooling, saline taste. It slowly effloresces on exposure to dry air.

Soluble in 1.1 parts of water at 25° C. (77° F.) and in 0.4 part of boiling water; slightly soluble in alcohol.

When heated to about 150° C. (302° F.) the salt loses all of its water of crystallization; on ignition at a red heat it decomposes and a mixture of sodium carbonate and carbon is left, which has an alkaline reaction and strongly effervesces with acids. To a non-luminous flame the salt imparts an intense yellow color.

An aqueous solution of Sodium Citrate (1 in 20) is slightly alkaline to red litmus-paper.

Tests for Identity.—See Sodium and Citrate in Part V.

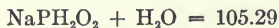
Impurities.—Carbonate and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This new official is made by treating sodium carbonate with citric acid. It, like the other alkaline citrates, has the property of holding in solution such insoluble bodies as ferric phosphate and magnesium carbonate. It is used as a refrigerant.

Dose.—1 Gm. (15 grains).

SODII HYPOPHOSPHIS. Sodium Hypophosphite.



It should contain not less than 98 per cent. of pure Sodium Hypophosphite $[\text{PO.H}_2\text{ONa} + \text{H}_2\text{O}]$ and should be kept in well-stoppered bottles; caution should be observed in dispensing Sodium Hypophosphite, as explosion is liable to occur when it is triturated or heated with nitrates, chlorates, or other oxidizing agents.

Small, colorless, transparent, rectangular plates of a pearly lustre, or a white, granular powder, odorless, and having a bitterish-sweet, saline taste. Very deliquescent on exposure to moist air.

Soluble in about 1 part of water and in about 25 parts of alcohol at 25° C. (77° F.); in 0.12 part of boiling water and in 1 part of boiling alcohol; slightly soluble in absolute alcohol; insoluble in ether.

When heated in a test-tube, the salt at first loses its water of crystallization, and at about 200° C. (392° F.) it is decomposed, evolving hydrogen and hydrogen phosphide, which burns spontaneously with a bright yellow flame. Finally, there is left a residue of sodium pyrophosphate and metaphosphate, sometimes mingled with a little red phosphorus. To a non-luminous flame the salt imparts an intense yellow color.

An aqueous solution of Sodium Hypophosphite is neutral or slightly alkaline to litmus-paper.

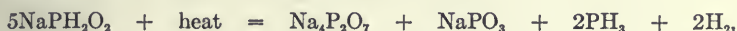
Tests for Identity.—See Sodium and Hypophosphites in Part V.

Impurities.—Caustic alkali, carbonate, heavy metals, and arsenic. Details in Part V.

This hypophosphite is prepared in a manner similar to that for

making potassium hypophosphite—by treatment of calcium hypophosphite with sodium carbonate.

Sodium hypophosphite occurs in the form of small transparent plates, or chiefly in white, granular powder. It is soluble in 1 part of water and 30 parts of alcohol. Like all hypophosphites, the sodium hypophosphite must be heated with great care, as otherwise the following chemical reaction will occur:



the resulting phosphine (PH_3) being spontaneously combustible and producing an explosion.

Sodium hypophosphite is a constituent of the official, *syrup of hypophosphites* and *compound syrup of hypophosphites*. It may be here stated that hypophosphorous acid and hypophosphites act as reducing agents on silver and mercury compounds, hence the salts of these metals should not be prescribed with syrup of hypophosphites. Like all hypophosphites, the salt of sodium is used as a tonic and nutrient in wasting diseases.

Dose.—1 Gm. (15 grains).

SODII IODIDUM. Sodium Iodide.

$\text{NaI} = 148.78$

It should contain not less than 98 per cent. of pure Sodium Iodide and should be kept in well-stoppered bottles.

Colorless, cubical crystals, or a white, crystalline powder; odorless and having a saline and slightly bitter taste. In moist air Sodium Iodide deliquesces and frequently undergoes decomposition, the salt assuming a brown tint.

Soluble in about 0.5 part of water and in about 3 parts of alcohol at 25°C . (77°F .); in 0.33 part of boiling water and in 1.4 part of boiling alcohol.

When strongly heated the salt melts, and at a bright red heat it is slowly volatilized and partly decomposed. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution of Sodium Iodide is slightly alkaline to red litmus-paper.

Tests for Identity.—See Sodium and Iodides in Part V.

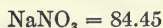
Impurities.—Excess of alkali, excess of potassium, heavy metals, free iodine, excess of iodate, barium, cyanide, excess of nitrates and nitrites, excess of chlorides or bromides, thiosulphate. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt so closely resembles potassium iodide that little additional statement is needed here. It is made by the same processes used in the manufacture of potassium iodide and the bromides of potassium and sodium. Hence here it is only necessary to mention that treating sodium hydroxide with iodine, and subsequent heating of the evaporated mass with charcoal, will yield the iodide.

It occurs in anhydrous cubes or rhombic prisms, somewhat deliquescent, turning reddish-brown on exposure to air, the latter change being due to liberation of iodine. It is used medicinally exactly as is potassium iodide.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

SODII NITRAS. Sodium Nitrate.

It should contain not less than 99 per cent. of pure Sodium Nitrate $[\text{NO}_3.\text{ONa}]$ and should be kept in well-stoppered bottles.

Colorless, transparent, rhombohedral crystals, odorless, and having a cooling saline, and slightly bitter taste. Hygroscopic in moist air.

Soluble in about 1.1 parts of water and in about 100 parts of alcohol at 25° C. (77° F.); in 0.6 part of boiling water and in 40 parts of boiling alcohol.

When heated to 312° C. (593.6° F.) the salt melts without decomposition. At a higher temperature it evolves oxygen and is reduced to nitrite. When Sodium Nitrate is heated with charcoal, the mixture deflagrates. To a non-luminous flame it imparts an intense yellow color.

Its aqueous solution is neutral to litmus-paper.

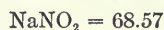
Tests for Identity.—See Sodium and Nitrates in Part V.

Impurities.—Heavy metals, iodides. Details in Part V.

This chemical is a purified form of the native *Chile salt-peter*. Large deposits of this mineral are found in Peru, and is called Chile salt-peter because it is shipped through the Chilean ports. The native ore is a combination of sodium nitrate with sodium iodate, and the latter chemical, as mentioned on p. 395, yields practically all the iodine in use in the world. The iodate is removed from the nitrate by crystallization in the district where mined, and is there worked into iodine (p. 396), while the nitrate is shipped into commerce practically free from iodide.

Sodium nitrate is official because it is the source of the commercial nitrates, it being rarely used in medicine, the potassium nitrate being preferred. When used, it is as a diuretic, as is potassium nitrate.

Dose.—1 Gm. (15 grains).

SODII NITRIS. Sodium Nitrite.

It should contain not less than 90 per cent. of pure Sodium Nitrite $[\text{NO}.\text{ONa}]$ and should be kept in well-stoppered bottles.

White, opaque, fused masses, or pencils, or colorless, transparent, hexagonal crystals; odorless and having a mild, saline taste.

When exposed to the air the salt deliquesces and is gradually oxidized to sodium nitrate, and becomes unfit for use.

Soluble in about 1.4 parts of water at 25° C. (77° F.) and very soluble in boiling water; slightly soluble in alcohol.

When heated the salt melts, and at a red heat it is decomposed, yielding oxygen, nitrogen, nitrogen dioxide, and sodium oxide. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution of Sodium Nitrite should be colorless and give a slightly alkaline reaction with red litmus-paper.

Tests for Identity.—See Sodium and Nitrites in Part V.

Impurities.—Heavy metals. Details in Part V.

Assay.—Details in Part V.

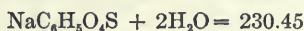
Remarks.—This salt is made by reduction of sodium nitrate by fusing with sheet lead. The fused mass is poured into water, the diluted solution treated with nitric acid until just neutral (thus precipitating the lead). The filtrate is then concentrated, when the sodium nitrite crystallizes out.

This salt occurs in the form of deliquescent granules, or usually in the form of fused pencils. It was introduced in the pharmacopœia

in 1890, because used in the manufacture of spirit of nitrous ether, and has been retained in the present pharmacopœia for the same reason. It is practically never used in medicine, although at one time the writer had a large demand for a mixture of it and sodium metaborate as a remedy for diabetes.

Dose.—65 milligrammes (1 grain).

SODII PHENOLSULPHONAS. Sodium Phenolsulphonate.



[SODII SULPHOCARBOLAS, PHARM. 1890]

It should contain not less than 99 per cent. of pure Sodium Paraphenolsulphonate [$\text{C}_6\text{H}_4(\text{OH})\text{SO}_3\text{Na} : 1.4 + 2\text{H}_2\text{O}$] and should be kept in well-stoppered bottles.

Colorless, transparent, rhombic prisms; odorless and having a cooling, saline, bitter taste. Somewhat efflorescent in dry air.

Soluble in 4.8 parts of water and in about 130 parts of alcohol at 25° C. (77° F.); in 0.7 part of boiling water and in 10 parts of boiling alcohol.

When heated a little above 100° C. (212° F.) the salt loses all of its water of crystallization (15.5 per cent.) and becomes white. At a higher temperature it chars, emits inflammable vapors having the odor of phenol, and finally leaves a residue of sodium sulphate amounting to 30.6 per cent. of the original weight. To a non-luminous flame it imparts an intense yellow color.

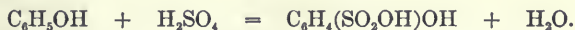
An aqueous solution of Sodium Phenolsulphonate is neutral to litmus-paper.

Tests for Identity.—See Sodium and Phenolsulphonate in Part V.

Impurities.—Heavy metals. Details in Part V.

Remarks.—This body, formerly called *sodium sulphocarbolate*, has a complex formula, $\text{NaSO}_3\text{C}_6\text{H}_4\text{OH}$. This is the sodium salt of sulphocarbolic acid, which is made by treating phenol (or carbolic acid) with sulphuric acid, whereby one hydrogen of $\text{C}_6\text{H}_5\text{OH}$ (see graphic formula on p. 768) is replaced by the group SO_2OH , which will be recognized as sulphuric acid, less one hydroxyl. This union gives us $\text{C}_6\text{H}_4\text{—}\begin{smallmatrix} \text{SO}_2\text{OH} \\ \text{OH} \end{smallmatrix}$ sulphocarbolic acid, and the sodium in the salt replaces the hydrogen of the hydroxyl attached to the SO_2OH group.

Sodium sulphocarbolate is made by treating phenol with sulphuric acid, and letting stand for twenty-four hours, when sulphocarbolic acid is formed:



To the acid mixture barium carbonate is added, thus forming barium sulphocarbolate, which is soluble, while the excess of sulphuric acid separates out as barium sulphate. To the filtered solution of barium sulphocarbolate sodium carbonate is added, producing the following reaction:



The barium carbonate thus formed precipitates out and is separated by filtration from the solution of sodium sulphocarbolate, which is then concentrated by evaporation to a point where crystallization takes place.

Sodium sulphocarbolate occurs as transparent, colorless prisms, somewhat efflorescent, possessing a delicate pink tint. This color-

tion, however, is due to the same cause as is the pink color of phenol, and hence may possibly be considered as an impurity.

Sodium sulphocarbolate is used as an antiferment.

Dose.—250 milligrammes (4 grains).

SODII PHOSPHAS. Sodium Phosphate.



It should contain, in an uneffloresced condition, not less than 99 per cent. of pure Di-sodium-ortho-phosphate $[\text{PO}(\text{OH})(\text{ONa})_2 + 12\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles, in a cool place.

Large, colorless, monoclinic prisms, or a granular, crystalline salt; odorless and having a cooling, saline taste. The crystals effloresce in the air, and gradually lose 5 molecules of their water of crystallization (25.1 per cent.).

Soluble in about 5.5 parts of water at 25° C. (77° F.); insoluble in alcohol.

When heated to about 40° C. (104° F.) the salt fuses, yielding a colorless liquid. At 100° C. (212° F.) it loses all its water of crystallization (60.3 per cent.), and at a red heat it is converted into sodium pyrophosphate. It imparts to a non-luminous flame an intense yellow color.

An aqueous solution of Sodium Phosphate is slightly alkaline to red litmus- and phenolphthalein-paper.

No residue should be left on dissolving the salt in water (absence of *calcium*, etc.).

Tests for Identity.—See Sodium and Phosphates in Part V.

Impurities.—Calcium, etc. (see above), heavy metals, carbonate, and arsenic. Details in Part V.

The formula of sodium phosphate is worthy of special notice. On p. 373 will be found the graphic formula of phosphoric acid, H_3PO_4 . If two of the three hydrogen atoms of phosphoric acid are replaced by sodium atoms (each atom of sodium of valence 1 replacing one atom of hydrogen), we get Na_2HPO_4 , the formula of the official sodium phosphate.

All these hydrogen atoms found in phosphoric acid are replaceable under proper conditions, as illustrated in the formula of official calcium phosphate, $\text{Ca}_3(\text{PO}_4)_2$ (p. 375), and a sodium phosphate having the formula Na_3PO_4 can be made. But the stable form of sodium phosphate is the one in which one atom of the hydrogen in the acid remains (Na_2HPO_4), and this is recognized by the pharmacopœia.

This important salt is prepared by treating bone-ash with sulphuric acid, when acid calcium phosphate is produced, as shown in the following equation:



The solution of acid calcium phosphate, freed from the insoluble calcium sulphate by forcible straining, treated with sodium carbonate, yields sodium phosphate, as shown in the following equation:



Other authorities say the reaction runs:



Sodium phosphate occurs in the form of large, colorless, efflorescent crystals, although now usually dispensed in the form of granules.

It is important for the pharmacist to remember that the granules are efflorescent, that they part comparatively easily with a portion of their water of crystallization, and thus lose a considerable amount of their weight. In fact, in the writer's experience, he has known of cases where a pound of sodium phosphate decreased to 12 ounces by weight by simple efflorescence. In selling the salt this should be kept in mind, otherwise a depreciation in the profit will result.

Recent literature has shown that a large amount of sodium phosphate on the market is contaminated with arsenic, and therefore the pharmacist should examine his phosphate in order to prevent dispensing a dangerous chemical. An interesting preparation of the salt is a combination of it with sodium nitrate and citric acid. When these three chemicals are triturated together in a warm mortar, they liquefy, forming a solution containing over 100 per cent. by volume of sodium phosphate. This is now official as *compound solution of sodium phosphate* (p. 472).

Medical Properties.—A valuable saline purgative.

Dose.—2 Gm. (30 grains).

SODII PHOSPHAS EFFERVESCENS. Effervescent Sodium Phosphate.

<i>Recipe.</i> —Exsiccated Sodium Phosphate, in fine powder, two hundred grammes.....	200 Gm.
Sodium Bicarbonate, dried and powdered, four hundred and seventy-seven grammes.....	477 Gm.
Tartaric Acid, dried and powdered, two hundred and fifty-two grammes.....	252 Gm.
Citric Acid, uneffloresced crystals, one hundred and sixty-two grammes.....	162 Gm.
To make about one thousand grammes..	1000 Gm.

Powder the Citric Acid and mix it intimately with the Exsiccated Sodium Phosphate and Tartaric Acid, then thoroughly incorporate the Sodium Bicarbonate.

Place the mixed powders on a plate of glass or in a suitable dish, in an oven heated to between 93° and 104° C. (199.4° and 219.2° F.).

When the mixture has acquired a moist consistence, by the aid of careful manipulation with a wooden spatula, rub it through a No. 6 tinned-iron sieve, and dry the granules at a temperature not exceeding 54° C. (129.2° F.). Keep the product in well-stoppered bottles.

Remarks.—This new official is a granular effervescent salt containing 20 per cent. exsiccated sodium phosphate. Note that the pharmacopœia directs that the mass be softened for granulation by warming and not by moistening with alcohol. It is a useful and agreeable purgative.

Dose.—8 Gm. (120 grains).

SODII PHOSPHAS EXSICCATUS. Exsiccated Sodium Phosphate.

It should contain not less than 99 per cent. of pure anhydrous Sodium Phosphate [$\text{PO}(\text{OH})(\text{ONa})_2$] and should be kept in well-stoppered bottles.

Sodium Phosphate, a convenient quantity.

Allow the crystals to effloresce for several days in warm air, at a temperature of from 25° to 30° C. (77° to 86° F.), then continue the drying in an oven; raise the temperature very gradually until 100° C. (212° F.) has been reached, and main-

tain this temperature until the salt ceases to lose weight. Powder and sift the residue and preserve it in well-stoppered bottles.

A white powder which absorbs moisture readily, and conforms to the reaction and tests given under *Sodii Phosphas*.

Remarks.—This new official is used for the manufacture of the granular effervescent salt just described. Note that its dose is just half of that of the crystalline salt.

Dose.—1 Gm. (15 grains).

LIQUOR SODII PHOSPHATIS COMPOSITUS. Compound Solution of Sodium Phosphate.

<i>Recipe.</i> —Sodium Phosphate, uneffloresced crystals, one thousand grammes.....	1000 Gm.
Sodium Nitrate, forty grammes.....	40 Gm.
Citric Acid, one hundred and thirty grammes.....	130 Gm.
Distilled Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

Triturate the Sodium Phosphate and Sodium Nitrate, in a mortar, with the Citric Acid, until completely liquefied, then add sufficient Distilled Water to make the product measure *one thousand cubic centimeters*. Filter the liquid. Keep the Solution in well-stoppered bottles in a moderately warm place.

Tests for Identity.—See Phosphates, Nitrates, and Citrates in Part V.

Remarks.—This preparation was originally introduced as a proprietary called “*melachol*.”

It has the advantage of being a very concentrated (100 per cent.) solution of sodium phosphate.

Dose.—8 Cc. (2 fluidrachms).

SODII PYROPHOSPHAS. Sodium Pyrophosphate.



It should contain, in an uneffloresced condition, not less than 99 per cent. of pure Sodium Pyrophosphate $[(\text{PO})_2\text{O}(\text{ONa})_4 + 10\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms, or a crystalline powder; odorless and having a cooling, saline, and feebly alkaline taste; slightly efflorescent in warm air.

Solution in about 11.5 parts of water at 25° C. (77° F.) and in 1.1 parts of boiling water; insoluble in alcohol.

When heated to 100° C. (212° F.) the salt loses its water of crystallization (40.35 per cent.) without previous fusion. At a higher temperature it fuses, forming a transparent liquid, which, on cooling, solidifies to a crystalline mass. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution is feebly alkaline to red litmus and to phenolphthalein paper.

Tests for Identity.—See Sodium and Phosphates in Part V.

Impurities.—Orthophosphate, carbonate, heavy metals, and arsenic. Details in Part V.

Sodium pyrophosphate, $\text{Na}_4\text{P}_2\text{O}_7$, introduces us to an acid of phosphorus not yet considered—pyrophosphoric. On a preceding page (p. 459), under Borax, mention was made of the fact that the prefix “pyro-” indicates application of strong heat; this applies to pyrophosphoric acid.

On p. 373 the difference between true orthophosphoric acid, H_3PO_5 , the so-called orthophosphoric acid, H_3PO_4 , and metaphosphoric acid, HPO_3 , is graphically demonstrated, and mention is there made that all phosphoric acids are derived from phosphoric oxide, P_2O_5 , a body in which phosphoric acid acts with valence v.

If to P_2O_5 we add one molecule of water, we get metaphosphoric acid, viz.,



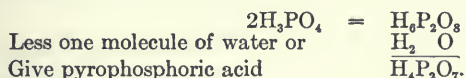
and if we add three molecules of water to one molecule of phosphoric oxide, we get the official phosphoric acid, viz.,



Likewise, if we subtract one molecule of water from official phosphoric acid, we get metaphosphoric acid, viz.,



If, on the other hand, we subtract one molecule of water from two molecules of official phosphoric acid, we get pyrophosphoric acid, as shown below:

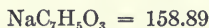


The pyrophosphate is obtained by heating sodium phosphate until its solution no longer gives a yellow precipitate with silver nitrate.

Sodium pyrophosphate occurs in the form of a crystalline powder or in monoclinic prisms, and is soluble in 12 parts of water. It is rarely employed in medicine, and is given place in the pharmacœia because required for making ferric pyrophosphate.

Dose.—2 Gm. (30 grains).

SODII SALICYLAS. Sodium Salicylate.



It should contain not less than 99.5 per cent. of pure Sodium Salicylate [$\text{C}_7\text{H}_4(\text{OH})\text{COONa}$] and should be kept in well-stoppered bottles, protected from heat and light.

White, microcrystalline powder or scales, or an amorphous, colorless powder, or having not more than a faint pink tinge; odorless and having a sweetish, saline taste.

Soluble in 0.8 part of water and in 5.5 parts of alcohol at 25°C . (77°F .); very soluble in boiling water or alcohol; also soluble in glycerin.

When heated the salt is decomposed, giving off inflammable vapors and an odor of phenol, and finally leaves a residue of sodium carbonate. To a non-luminous flame it imparts an intense yellow color.

An aqueous solution of Sodium Salicylate, when freshly made, should be colorless, and should slightly redden blue litmus-paper.

Tests for Identity.—See Sodium and Salicylates in Part V.

Impurities.—Sulphites and heavy metals. Details in Part V.

Assay.—Details in Part V.

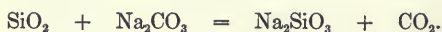
Remarks.—This salt is prepared by treating salicylic acid with sodium carbonate, and evaporating the resulting solution of sodium salicylate to dryness in the water-bath. Many modern therapeutists claim that the salicylic acid employed should be that from oil of wintergreen, and calls for "Sodium salicylate (natural)" can be easily filled by the retail pharmacist by treating oil of wintergreen with the molecular quantity of sodium hydroxide, evaporating the mass to dryness, redissolving in water, making the solution acid by the addition of more salicylic acid, and granulating the resulting filtered liquid. The addition of extra acid is essential, for the alkaline sodium salicylate is invariably of grayish color. In preparing the sodium salicylate great care must be taken to avoid contact with iron, as that element will color the salicylate a blood red. (See p. 783.)

Sodium salicylate is a valuable remedy for rheumatism.

Dose.—1 Gm. (15 grains).

Sodium silicate, Na_2SiO_3 , was recognized in the pharmacopœia of 1890 as a nearly saturated solution (*liquor sodii silicatis* or *liquid glass*). It is made by fusing together common sand and sodium carbonate in the presence of a small quantity of charcoal. The fused mass is poured into water in order to obtain the product in small porous masses, which are more readily soluble.

Sand is silicic oxide, SiO_2 , an exact analogue to carbonic oxide, CO_2 . Silicic oxide plus sodium carbonate gives us sodium silicate and gaseous carbonic oxide:



Solution of sodium silicate is a more or less transparent, but faintly yellowish, viscid liquid, the keeping of which is a source of considerable annoyance to the retail pharmacist. If kept in glass-stoppered bottles it will cause the stopper to stick to the neck. It usually is somewhat alkaline, and therefore attacks cork, and if not tightly corked, it will comparatively easily evaporate to a glassy mass, which it is quite difficult to redissolve. This body is called *liquid glass* or *water glass*, because of its resemblance to ordinary glass, which is a combination of sodium silicate with the silicates of lead and calcium.

Liquid glass forms, on drying, a vitreous coating which is hard and inflexible. Accordingly, it is used in mending fractured limbs, as is also plaster of Paris, the broken member being swathed with bandages which are fixed firmly in their place by applications of the liquid glass. After standing, the solution solidifies to a hard, rigid mass, which securely and firmly encases the limb.

It is superior to plaster of Paris in the greater ease with which the glass dressing can be removed after the limb is set.

The plaster has to be chipped off—a very tiresome operation; the glass, on the other hand, can be dissolved off with hot water.

It is found, however, that liquid glass sets too slowly for the average operation, and plaster-of-Paris settings are, therefore, usually preferred.

SODII SULPHAS. Sodium Sulphate.



It should contain, in an effloresced condition, not less than 99 per cent. of pure Sodium Sulphate [$\text{SO}_4(\text{ONa})_2 + 10\text{H}_2\text{O}$] and should be kept in well-closed vessels, in a cool place.

Large, colorless, transparent, monoclinic prisms, or granular crystals; odorless, and having a bitter, saline taste. The salt effloresces rapidly in the air and, finally, loses all of its water of crystallization.

Soluble in 2.8 parts of water at 15°C . (59°F .), in 0.25 part at 34°C . (93.2°F .), and in 0.47 part at 100°C . (212°F .); insoluble in alcohol; soluble in glycerin.

When heated to 33°C . (91.4°F .) the salt fuses, and, on continuing the heat to 100°C . (212°F .) it loses all of its water of crystallization (55.9 per cent.). At

a red heat the anhydrous salt fuses without decomposition. To a non-luminous flame it imparts an intense yellow color.

Its aqueous solution is neutral to litmus-paper.

Tests for Identity.—See Sodium and Sulphates in Part V.

Impurities.—Heavy metals and arsenic. Details in Part V.

Sodium sulphate is known by the synonym of *Glauber's salt*, so named after John Rudolph Glauber, who published the process of manufacture in his work, "*De Natura Salium*," 1658. It is made by treating sodium chloride with sulphuric acid, and is, therefore, an intermediate product in the Leblanc process (p. 461).

Native sodium sulphate had been used many years before this.

Sodium sulphate occurs in efflorescent, monoclinic prisms or granular crystals, possessing a bitter, saline taste. Its solubility in water is noteworthy, the following table showing its variations from 0 to 100° C.:

At 0° C.	1 part crystalline sodium sulphate dissolves in 8 parts of water.
At 15° C.	" " " " " " " 2.8 " " "
At 25° C.	" " " " " " " 1 part " "
At 30° C.	" " " " " " " 0.5 " " "
At 34° C.	" " " " " " " 0.25 " " "
At 50° C.	" " " " " " " 0.37 " " "
At 100° C.	" " " " " " " 0.40 " " "

the temperature being, of course, that of the water.

By this table it will be noted that the maximum solubility is reached at 34° C., when one part of the salt (crystals) will dissolve in one-quarter its weight of water. At 35° C. the solubility is 1 part in 0.3 part water, and from that to 100° the solubility slowly decreases. This phenomenon has been explained by claiming that up to 34° C. we deal with solution of the salt containing ten molecules of water of crystallization. At 34° it is supposed that the salt loses nine molecules of water, and from that time on we deal with a chemical possessing only one molecule of water of crystallization. The solution of sodium sulphate at 34° is a striking illustration of a supersaturated solution. Such a solution will remain intact if kept in a quiet place, even though the temperature of the solution be reduced below 33°. If the flask is shaken, or even if the cork is removed, there will be a sudden formation of crystals, with the evolution of considerable heat, corresponding to the phenomenon of *retarded crystallization*. (See p. 122.) The crystallization due to agitation is easy to understand. One explanation of why the crystallization takes place by merely removing the cork is that the air contains minute crystals of sodium sulphate, which, when the cork is removed, fall into the solution and act as nuclei.

Sodium sulphate is a useful purgative.

Dose.—16 Gm. (240 grains).

SODII SULPHIS. Sodium Sulphite.



It should contain, in the uneffloresced and air-dried condition, not less than 96 per cent. of pure Sodium Sulphite, and should be kept in well-stoppered bottles, in a cool place.

PRINCIPLES OF PHARMACY

transparent, monoclinic prisms; odorless and having a cooling, saline, ste. Exposed to the air the salt effloresces, and is slowly oxidized

soluble in 2 parts of water at 25° C. (77° F.) and in 1.4 parts of boiling water; sparingly soluble in alcohol.

When gently heated the salt softens somewhat, but it does not fuse. Above 100° C. (212° F.) the crystals lose all their water (50 per cent.), without fusing or changing their shape. At a red heat the salt fuses to a reddish-yellow mass of sodium sulphate and sodium sulphide. To a non-luminous flame the salt imparts an intense yellow color.

Its aqueous solution is neutral or feebly alkaline to litmus-paper.

Tests for Identity.—See Sodium and Sulphites in Part V.

Impurities.—Thiosulphate and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This chemical is made by passing sulphur dioxide gas, produced in the way as explained under sulphurous acid (p. 424), into a solution of sodium carbonate, and concentrating the solution to a point until it gives the separation of the crystals.

Sodium sulphite appears in efflorescent, monoclinic crystals, having no odor and possessing a saline, sulphurous taste. On standing, the salt slowly oxidizes to sodium sulphate. Sodium sulphite is used as an antiferment.

Dose.—1 Gm. (15 grains).

SODII THIOSULPHAS. Sodium Thiosulphate.



[SODII HYPOSULPHIS, PHARM. 1890]

It should contain not less than 98 per cent. of pure Sodium Thiosulphate and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms; odorless and having a cooling, afterward bitter taste. Permanent in the air below 33° C. (91.4° F.), but efflorescent in dry air above that temperature; slightly deliquescent in moist air.

Soluble in about 0.35 part of water at 25° C. (77° F.); at a boiling heat the solution is rapidly decomposed; insoluble in alcohol; slightly soluble in oil of turpentine.

When rapidly heated to 50° C. (122° F.) the salt melts. When slowly heated until it is effloresced, and afterward to 100° C. (212° F.), it loses all of its water of crystallization (36.3 per cent.), and at a red heat is decomposed, sulphur being liberated, while a residue of sodium sulphide and sodium sulphate remains. To a non-luminous flame it imparts an intense yellow color.

Its aqueous solution is neutral or faintly alkaline to litmus-paper.

Tests for Identity.—See Sodium and Thiosulphate in Part V.

Impurities.—Sulphites and bisulphites, arsenic, heavy metals, calcium, caustic alkali and carbonates, sulphide. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt has been incorrectly called sodium hypsulphite. In the table of valences on p. 367 it will be found that the valence of sulphur is II, IV, and VI.

As already learned, when an element has three valences and three oxides, the oxide showing the highest valence is given the termination "ic," while the oxide showing the lowest valence has "hypo" prefixed to the name of the positive element, whose termination becomes "ous." Under this rule, that oxide in which sulphur acts with the

valence II should be termed hyposulphurous oxide, and the formula of this body is SO.

If hyposulphurous oxide be SO, then hyposulphurous acid will be $\text{SO} + \text{H}_2\text{O}$ or SO_2H_2 , and sodium hyposulphite will be Na_2SO_2 , which is quite different from the formula given above; the so-called sodium hyposulphite, $\text{Na}_2\text{S}_2\text{O}_3$, being really sodium thiosulphate. "*Thios*" is Greek for sulphur, hence the literal translation of thiosulphate is "sulphosulphate," a most appropriate title for the body $\text{Na}_2\text{S}_2\text{O}_3$, as is shown by the following comparison of the graphic formula of it with that of normal sodium sulphate:

SODIUM SULPHATE.



SODIUM THIOSULPHATE.



Sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3$, can be resolved into Na_2SO_3 (sodium sulphite) and sulphur, and inversely the thiosulphate can be made by boiling the sulphite with sulphur.

The commercial method of manufacture is by treating calcium thiosulphate with sodium sulphate, as shown in the following equation:



The calcium thiosulphate used in the above reaction is obtained from the calcium sulphide residue of the Leblanc process (p. 462). On exposure to air the calcium sulphide found therein is oxidized to calcium thiosulphate, which is lixiviated from the insoluble matter of the residue. Sodium thiosulphate is also made by cooking sulphur with sodium sulphate.

Sodium thiosulphate occurs in colorless, transparent crystals, soluble in less than one part of water. It is used largely in commerce, being a valuable antichlor in paper manufacture. It is the "hypo" of photographers, being used by them as a developer (see p. 621), it having the power of dissolving the chloride and bromide of silver. When brought in contact with an iodine solution, it immediately discharges the color of same, as explained on p. 396. The decolorizing influence of sodium thiosulphate on iodine is applied in the estimation of the iodine strength of various pharmaceuticals, the same being titrated with decinormal solution of sodium thiosulphate. Sodium thiosulphate has been recommended as exhibiting all the therapeutic value of sulphur in a soluble form, it being stated, when a solution enters the stomach, the acids thereof throw out a sufficient amount of sulphur to become active. In the same way the ointment of sodium hyposulphite has been recommended in place of sulphur ointment. When given internally, its dose is 10 to 20 grains.

CHAPTER XXVII

LITHIUM AND AMMONIUM

LITHIUM

Symbol, Li. Atomic weight, approximately 7.

THE word lithium is derived from the Greek "*lithios*," meaning "stone," being so named by Berzelius because found in minerals. The presence of the new element was first detected by Arfvedson in 1817, but its isolation in more than traces was not accomplished until 1855, when Bunsen and Mattiessen obtained it by electrolysis of fused lithium chloride.

The chief ores of lithium are *lepidolite*, a combination of the silicates of lithium, sodium, and aluminum, and *triphyline*, which is a mixture of the phosphates of lithium, sodium, iron, manganese, and calcium.

It was first discovered in mineral water by Berzelius (1822), who noted its presence in Carlsbad water.

It is also found in tobacco and in the sugar-beet.

The metal cannot be isolated by chemical means, such as are used in obtaining sodium and potassium, by reason of the non-volatile character of the metal, but is prepared only by electrolysis of the chloride.

Lithium is the lightest of all metals, having the specific gravity 0.59, *i. e.*, it has less than six-tenths the weight of water, and is lighter than petroleum or ether, and hence floats on both of these liquids. It is a soft, silvery-white metal, possessing characteristics similar to that of the other members of the group, sodium and potassium, although being less chemically active. Thus, while it reacts in water with the formation of lithium hydroxide, it does not do so with explosive violence that characterizes the same reaction in the case of sodium and potassium.

The most characteristic test for lithium is the crimson color which it or its salts transmits to a colorless flame. If the solution of the chloride be treated with ammonium carbonate, the rather sparingly soluble carbonate is precipitated. This carbonate is somewhat soluble in water, however (1 part in 75 parts water), hence the statement already made (p. 431) that the carbonates of the alkalis dissolve in water still obtains.

The following salts of lithium and their preparations are official:

Lithium benzoate, 98.5 per cent. absolute $\text{LiC}_7\text{H}_5\text{O}_2$.
 Lithium bromide, 97 per cent. absolute LiBr .
 Lithium carbonate, 98.5 per cent. absolute Li_2CO_3 .
 Lithium citrate, 98.5 per cent. absolute $\text{Li}_3\text{C}_6\text{H}_5\text{O}_7$ (when dried).
 Effervescent lithium citrate contains 5 per cent. of lithium citrate.
 Lithium salicylate, 98 per cent. absolute $\text{LiC}_7\text{H}_5\text{O}_2$.

LITHII BENZOAS. Lithium Benzoate.

$\text{LiC}_7\text{H}_5\text{O}_2 = 127.11$

It should contain not less than 98.5 per cent. of pure Lithium Benzoate [$\text{C}_6\text{H}_5\text{-COOLi}$] and should be kept in well-stoppered bottles.

A light, white powder, or small, shining, crystalline scales; odorless, or of faint benzoïn-like odor, and of a cooling, sweetish taste; permanent in the air.

Soluble in 3 parts of water and in 13 parts of alcohol at 25° C. (77° F.); in 2.5 parts of boiling water and in 10 parts of boiling alcohol. Its solubility in water is increased by the presence of sodium benzoate, but lessened when alcohol is the solvent.

When heated the salt fuses; at a higher temperature it chars, emits inflammable vapors having a benzoïn-like odor, and finally leaves a residue of lithium carbonate mixed with carbon. This residue imparts a crimson color to a non-luminous flame, and its aqueous solution has an alkaline reaction upon litmus-paper.

The aqueous solution (1 in 20) of Lithium Benzoate has an alkaline reaction upon litmus, but is neutral to phenolphthaleïn T.S.

The addition of a few drops of ferric chloride T.S. to the above solution will produce a voluminous flesh-colored precipitate of ferric benzoate.

If an excess of hydrochloric acid be added to a concentrated aqueous solution of 0.6 Gm. of Lithium Benzoate, a white precipitate of benzoic acid will be formed, which, after collecting upon a filter and thoroughly washing and drying, should respond to the tests of purity given under *Acidum Benzoicum*.

If 1 Gm. of the salt be dissolved in 20 Cc. of water, and the benzoic acid precipitated by the addition of a sufficient quantity of hydrochloric acid, the filtrate should respond to the following tests of purity:

The addition of ammonia water until the solution has an alkaline reaction should produce neither turbidity nor precipitation, either before or after boiling (absence of iron, aluminum, etc.).

Tests for Identity. See Lithium and Benzoates, above.

Impurities.—Iron and aluminum, see above. Excess of alkali and heavy metals. See Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by treating lithium carbonate with benzoic acid, as shown in the following equation:



It occurs in a light white powder or small, shiny scales, and is used as a diuretic and as a remedy for gout.

Dose.—1 Gm. (15 grains).

LITHII BROMIDUM. Lithium Bromide.

LiBr = 86.34

It should contain when well dried not less than 97 per cent. of pure Lithium Bromide and should be kept in well-stoppered bottles.

A white, granular salt, odorless, and having a sharp, slightly bitter taste; very deliquescent.

Soluble in 0.6 part of water at 25° C. (77° F.) and in 0.3 part of boiling water; very soluble in alcohol; also soluble in ether.

At a low red heat the salt fuses, and at a higher heat it is slowly volatilized.

The aqueous solution is slightly alkaline to litmus-paper.

Tests for Identity.—See Lithium and Bromides in Part V.

Impurities.—Other alkalis, iron, and aluminum. See Lithium Benzoate. Iodine, excess of potassium, and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by treating lithium carbonate with potassium bromide, as shown in the following equation:

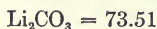


or by treating lithium sulphate with potassium bromide. In both cases the potassium salt—the carbonate or the sulphate—is precipitated from the lithium bromide solution by the addition of a small quantity of alcohol. Yvon's process of preparation is by treating lithium carbonate with bromine and then passing hydrogen sulphide through the mixture until the color of the bromine has disappeared. In this case the sulphide acts on the bromine to form hydrobromic acid and sulphur, the latter precipitating. The hydrobromic acid, in turn, acts on the lithium carbonate to form lithium bromide, and the result of the reaction is a solution of lithium bromide containing a precipitate of sulphur, which is removed by filtration. The filtered liquid is concentrated by evaporation and crystallized by desiccation, under the bell-glass, with sulphuric acid. A fourth process suggested is by treating ferrous bromide with lithium carbonate, and is, therefore, similar to the process for making potassium bromide. (See p. 439.)

Lithium bromide is a deliquescent, white, granular salt, having a slightly sharp, bitter taste, and soluble in 0.6 part of water. It is used as a hypnotic.

Dose.—1 Gm. (15 grains).

LITHII CARBONAS. Lithium Carbonate.



It should contain not less than 98.5 per cent. of pure Lithium Carbonate $[\text{CO}-(\text{OLi})_2]$ and should be kept in well-stoppered bottles.

A light, white powder, odorless, and having an alkaline taste; permanent in the air.

Soluble in 75 parts of water at 25° C. (77° F.), also in 140 parts of boiling water; much more soluble in water saturated with carbon dioxide; insoluble in alcohol; soluble in diluted acids with active effervescence.

At a low red heat the salt fuses; at a higher temperature it loses some of its carbon dioxide, and is partially converted into lithium oxide. Its solution in hydrochloric acid imparts a crimson color to a non-luminous flame.

The aqueous solution has an alkaline reaction upon litmus-paper.

If 1 Gm. of Lithium Carbonate be dissolved in 40 Cc. of diluted acetic acid, no insoluble residue should remain.

Tests for Identity.—See Lithium and Carbonates in Part V.

Impurities.—Iron and aluminum and other alkalis. See Lithium Benzoate. Heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—Lithium carbonate is prepared from lepidolite by making a paste of the finely powdered ore with concentrated sulphuric acid, calcining, and treating the residue with water. This throws out silicic acid, and converts the metals into sulphates, which go into solution. To the solution, potassium sulphate is added, when, on concentration, the aluminum crystallizes out as alum. To the remaining solution of the sulphates of sodium, potassium, and lithium, sodium carbonate is added, when crude lithium carbonate precipitates. This is purified by cooking with milk of lime, when a solution of lithium hydroxide results, and from this the carbonate is produced by use of sodium carbonate. Lithium carbonate is also made by adding the chloride to warm 20 per cent. ammonium car-

bonate solution, or to solution of ammonium carbonate containing ammonia water.

Lithium carbonate is used in gout because of the solvent power it possesses over uric acid. Gout is a disease caused by the formation of deposits of calcium urate in the various joints, and to relieve gout it is essential that these deposits be removed. For this purpose nothing has been found to act so satisfactorily as do the various lithium compounds.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

LITHII CITRAS. Lithium Citrate.



It should, when carefully dried, contain not less than 98.5 per cent. of pure Lithium Citrate $[\text{C}_6\text{H}_4(\text{OH})(\text{COOLi})_3]$ and should be kept in well-stoppered bottles.

A white powder, or colorless crystals, odorless, and having a cooling, faintly alkaline taste; deliquescent on exposure to moist air.

Soluble in about 2 parts of water at 25°C . (77°F .) and in 1.5 parts of boiling water; almost insoluble in alcohol or ether.

At a red heat the salt chars, emits inflammable vapors having a pungent odor, and finally leaves a black residue of lithium carbonate mixed with carbon. It imparts a crimson color to a non-luminous flame.

The aqueous solution is neutral to litmus-paper, and should not redden phenolphthalein T.S.

Tests for Identity.—See Lithium and Citrates in Part V.

Impurities.—Other alkalis, iron, and aluminum. See Lithium Benzoate. Heavy metals. Details in Part V.

Assay.—If 0.5 Gm. of anhydrous Lithium Citrate be cautiously ignited in a porcelain crucible, and if, after cooling, the residue be moistened with a few drops each of nitric and sulphuric acids and again cautiously ignited, repeating the operation until the residue becomes white and of constant weight, then the residue of lithium sulphate should weigh not less than 0.387 Gm. nor more than 0.394 Gm.

Remarks.—This salt is made by treating the lithium carbonate with citric acid, and crystallization of the citrate by evaporation of the solution. It occurs in the form of a white, deliquescent powder, possessing a feebly alkaline taste, and is soluble in two parts of water. Its use is the same as that of the carbonate, but has the advantage of possessing a less disagreeable taste, and is more easily tolerated by the stomach.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

LITHII CITRAS EFFERVESCENS. Effervescent Lithium Citrate.

<i>Recipe</i> .—Lithium Citrate, fifty grammes.....	50 Gm.
Sodium Bicarbonate, dried and powdered, five hundred and seventy grammes.....	570 Gm.
Tartaric Acid, dried and powdered, three hundred grammes.....	300 Gm.
Citric Acid, uneffloresced crystals, one hundred and ninety-five grammes.....	195 Gm.
To make about one thousand grammes..	1000 Gm.

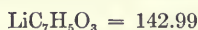
Powder the Citric Acid and mix it intimately with the Lithium Citrate and Tartaric Acid, then thoroughly incorporate the Sodium Bicarbonate. Place the mixed powders on a plate of glass or in a suitable dish, in an oven heated between 93° and 104°C . (199.4° and 219.2°F .).

When the mixture, by the aid of careful manipulation with a wooden spatula, has acquired a moist consistence, rub it through a No. 6 tinned-iron sieve, and dry the granules at a temperature not exceeding 54°C . (129.2°F .). Keep the product in well-stoppered bottles.

Remarks.—This effervescent salt is a 5 per cent. mixture of lithium citrate with sodium bicarbonate, dried tartaric acid, and crystalline citric acid, granulated by softening the mass with heat and rubbing through a coarse sieve.

Dose.—8 Gm. (120 grains).

LITHII SALICYLAS. Lithium Salicylate.



It should contain not less than 98.5 per cent. of pure Lithium Salicylate $[\text{C}_6\text{H}_4(\text{OH})\text{COOLi}]$ and should be kept in well-stoppered bottles.

A white or grayish-white powder, odorless, and having a sweetish taste; deliquescent in a moist atmosphere.

Very soluble in water and in alcohol.

When heated the salt is decomposed, emitting the odor of phenol, and finally leaving a residue of lithium carbonate and carbon. It imparts a crimson color to a non-luminous flame.

The aqueous solution slightly reddens blue litmus-paper.

The aqueous solution should be colorless (absence of *iron* and *organic coloring matters*), and should not effervesce upon the addition of diluted acids (absence of *carbonate*).

If copper sulphate T.S. be added to an aqueous solution of the salt (1 in 20), the mixture should have a bright green color.

Tests for Identity.—See Lithium and Salicylates in Part V.

Impurities.—Other alkalis, iron, and aluminum. See Lithium Benzoate. Organic coloring-matter and carbonate (see above). Heavy metals. Details in Part V.

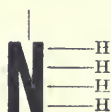
Assay.—If 0.5 Gm. of dry Lithium Salicylate be thoroughly mixed with about 1 Gm. of powdered anhydrous ammonium sulphate, and cautiously ignited in a porcelain crucible until of constant weight, the residue should weigh not less than 0.188 Gm. nor more than 0.192 Gm.

This is prepared by treating lithium carbonate with salicylic acid, heating until the effervescence ceases, and then granulating. The salt occurs in the form of a grayish-white, deliquescent powder, having a sweetish taste, very soluble in both alcohol and water. Lithium salicylate is a popular remedy for rheumatism and gout, since it possesses the antirheumatic properties of both salicylic acid and lithium.

Dose.—1 Gm. (15 grains).

AMMONIUM

Ammonium is discussed as an element, although we know it is a compound and have no proof that it exists in a free state. By ammonium we mean the radicle NH_4 . Graphically,



LITHIUM AND AMMONIUM

The discussion of radicles found on p. 633 will explain with one free bond, can act like a metal with one free bond.

The only inkling we have of the metallic character of ammonium and the possibility of its eventual isolation come from the fact that, by electrolysis of ammonium hydroxide in the presence of mercury, there is formed an amalgam of ammonium with mercury similar to that yielded by metallic sodium or potassium with the same liquid elements.

This amalgam, on being heated, splits into mercury, ammonia (NH_3), and hydrogen.

Do not confound *ammonium* with *ammonia*. Ammonia is a gas of sharp, penetrating odor, characteristic of spirit of hartshorn, having the formula NH_3 , or, graphically,



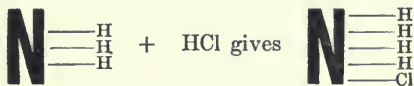
It is a definite chemical in which all three of the nitrogen bonds are satisfied by hydrogen. Ammonium is a radicle, showing nitrogen with five bonds and only four of these taken up by hydrogen, leaving one free bond ready for action. The formation of ammonium compounds from ammonia is different from metallic combinations. If we treat a metal—say, potassium—with hydrochloric acid, hydrogen is evolved and a chloride is formed, thus—



If, on the other hand, we treat ammonia with hydrochloric acid, ammonium chloride is formed without evolution of hydrogen, thus—



or, graphically,



This reaction seems to prove Frankland's theory concerning bonds, mentioned on p. 364.

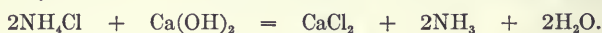
Again, let it be reiterated that *sodium* (Na) is not *soda* (NaOH), *potassium* (K) is not *potash* (KOH), and *ammonium* (NH_4) is not *ammonia* (NH_3).

Ammonia, as just mentioned, is a gas having the formula of NH_3 . This body is interesting as one of the few volatile alkalis (the others being organic chemicals), and its presence in compounds is generally detected by means of its volatility, the suspected substance being treated with soda or potassa and heated; if ammonium compounds are present, the ammonia gas is evolved, and is readily recognized by its characteristic odor. If only minute quantities of the gas are evolved, it can be detected by means of moistened red litmus-paper, which will turn blue upon being applied to the vapors of ammonia. When ammonium gas comes in contact with hydrochloric acid, white

fumes of ammonium chloride are formed. In testing for minute quantities of ammonia, Nessler's reagent is employed. This consists of a solution of mercuric iodide in an excess of mercuric chloride, and when this colorless or faintly straw-colored liquid is added to the solution containing ammonia, the liquid is then changed to either an amber tint or a red-brown precipitate is produced, according to the amount of ammonia present. By this means small quantities of ammonia (even 1 part to 10,000,000) can be detected in a liquid.

The test is too sensitive for application in ordinary analytic processes, but is invaluable in detecting ammonia in drinking-water, as already explained on p. 385.

Ammonia gas was first prepared by Priestly, in 1774, by heating ammonium chloride with slaked lime, the reaction being:



The evolved gas he called "alkaline air," the word ammonia (or Ammoniac) being first applied to it by Bergman in 1782.

Commercial ammonia gas is made from the ammoniacal liquor of gas-works. In the manufacture of illuminating-gas from coal the gas, as it leaves the retort, is passed through diluted sulphuric acid to remove the ammonia produced from the coal. In this way the acid liquid eventually becomes a solution of ammonium sulphate, and the crude sulphate obtained by evaporation of the solution is sold to the chemical manufacturers. By treating this crude sulphate with slaked lime the ammonia gas is evolved, and on being passed into water, forms the commercial solution of ammonia.

AQUA AMMONIÆ. Ammonia Water.

An aqueous solution of Ammonia [$\text{NH}_3=16.93$] containing 10 per cent., by weight, of gaseous ammonia. This solution deteriorates on keeping, and should be tested frequently. It must not be dispensed for medicinal purposes if it contains less than 10 per cent., by weight, of the gas. Ammonia Water should be kept in glass-stoppered bottles, in a cool place.

A colorless, transparent liquid, having a very pungent, characteristic odor, a caustic alkaline taste, and a strongly alkaline reaction upon red litmus-paper.

Specific gravity: 0.958 at 25° C. (77° F.).

It is completely volatilized at 100° C. (212° F.).

On bringing a glass rod dipped into hydrochloric acid near cold Ammonia Water, dense white fumes are produced.

On slightly supersaturating 10 Cc. of Ammonia Water with diluted sulphuric acid, no *empyreumatic odor* or *red color* should be developed, and if to this liquid 0.1 Cc. of tenth-normal potassium permanganate V.S. be added, the pink color should not be completely destroyed within ten minutes (absence of *readily oxidizable substances*).

If Ammonia Water be slightly supersaturated with nitric acid and be evaporated on a water-bath to dryness, it should be completely volatilized, leaving no residue on subsequent ignition (absence of *coal-tar bases* and of *fixed impurities*).

Tests for Identity.—White fumes with hydrochloric acid (see above).

Impurities.—Oxidizable substances, coal-tar bases (see above), carbonic acid, heavy metals, sulphates, chlorides. See Part V.

Assay.—Details in Part V.

Dose.—1 Cc. (15 minims).

AQUA AMMONIÆ FORTIOR. Stronger Ammonia Water.

An aqueous solution of Ammonia [$\text{NH}_3=16.93$] containing 28 per cent., by weight, of gaseous ammonia. This solution deteriorates on keeping, and should be tested frequently. Stronger Ammonia Water should be kept in partially filled, strong, glass-stoppered bottles, in a cool place. Great caution should be used in handling this liquid.

A colorless, transparent liquid, having an excessively pungent odor, a very caustic and alkaline taste, and a strongly alkaline reaction upon red litmus-paper. Specific gravity: 0.897 at 25° C. (77° F.).

If Stronger Ammonia Water be diluted with twice its volume of distilled water, it should respond to the reactions and tests described under *Aqua Ammonia*.

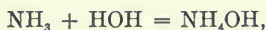
The percentage of ammonia gas in Stronger Ammonia Water should be determined by the same test as that given under *Aqua Ammonia*.

Remarks.—It will be seen that the pharmacopœia recognizes two strengths of ammonia water—the ordinary, containing 10 per cent., by weight, of ammonia gas, and the stronger water, which contains 28 per cent. of the same gas and represents a saturated solution. Unfortunately, as is usual with commercial chemicals, ammonia is not always sold by percentage of ammonia gas, but by the arbitrary marking of degrees.

The “degrees” mean the graduations of the Baumé scale of light liquids (see p. 49), and must not be confounded for the percentage. Thus, 17° ammonia, largely sold by wholesalers, does not contain 17 per cent. ammonia gas, but represents the specific gravity 0.953, which means only 11½ per cent. ammonia gas.

To render the subject still more vague, some wholesalers substitute for degrees Baumé arbitrary markings “F,” “FF,” “FFF,” and even “FFFF.” Presumably the first stands for “fortis” (strong), the second for “fortior” (stronger), and the third for “fortissimus” (strongest). In this case 4 F is outdoing the superlative.

When ammonia gas is run into water, ammonium hydroxide is produced by a reaction similar to that just cited as occurring between ammonia and hydrochloric acid. The reaction is:



the valence of nitrogen changing from III to V during the process.

This theory is disputed by Thomsen, who, from physical and thermic experiments, concluded that ammonia water is simply a solution of NH_3 gas in water.

Ammonia water (10 per cent.) is called *spirit of hartshorn* because originally made by the destructive distillation of the horn of the hart (*Cornu cervi*). Regarding the stronger ammonia water, containing 28 per cent. of ammonia gas, nothing need be added to what has already been said on p. 167. In addition to these two aqueous solutions of ammonia gas the pharmacopœia recognizes an alcoholic solution containing 10 per cent. by weight of ammonia gas; this is the official *spiritus ammonia*.

SPIRITUS AMMONIÆ. Spirit of Ammonia.

An alcoholic solution of Ammonia [$\text{NH}_3=16.93$] containing 10 per cent., by weight, of the gas. This solution deteriorates on keeping, and should be tested frequently. It must not be dispensed for medicinal purposes if it contains less than 10 per cent. of gaseous Ammonia.

Recipe.—**Stronger Ammonia Water**, two hundred and fifty cubic centimeters..... 250 Cc.
Alcohol, recently distilled, and after distillation kept in glass vessels, a sufficient quantity.

Pour the Stronger Ammonia Water into a flask provided with a safety funnel, and connected, by means of a glass condenser, with a well-cooled receiver containing five hundred cubic centimeters of Alcohol, the delivery tube of the condenser reaching nearly to the bottom of the receiver. Heat the flask carefully, and very gradually, to a temperature not exceeding 60° C. (140° F.), and maintain it at that temperature until Ammonia ceases to be evolved. Then disconnect the receiver, and, having ascertained the strength of a portion of the contents by the method of assay given below, add enough Alcohol to make the product contain *ten per cent.*, by weight, of Ammonia gas. Keep the Spirit in glass-stoppered bottles, in a cool place.

A colorless liquid, having a strong odor of ammonia, and a specific gravity of about 0.808 at 25° C. (77° F.).

When diluted with water, it should respond to the tests for identity and purity given under *Aqua Ammonia*.

Assay.—Details in Part V.

Remarks.—The pharmacopœial process of manufacture, by placing the stronger water of ammonia in a retort and driving off the ammonia gas by heat into alcohol, and finally estimating the ammonia strength of the alcohol, and reducing to 10 per cent. value, was found by the writer to be useless, his experience being that three times the pharmacopœial amount of ammonia water produced a spirit of ammonia containing but 7 per cent. of ammonia gas. In fact, the only way he succeeded in getting a 10 per cent. solution spirit of ammonia was by the production of ammonia gas by treating the chloride with an alkali, and passing this gas into alcohol under pressure.

Dose.—1 Cc. (15 minims).

AMMONIUM SALTS

Ammonium salts all contain the radicle NH_4 , which acts very much like the elements sodium and potassium. Thus, while the formula of sodium bromide is NaBr , so the formula of ammonium bromide is NH_4Br ; as the formula of potassium sulphate is K_2SO_4 , so that of ammonium sulphate is $(\text{NH}_4)_2\text{SO}_4$.

The following ammonium salts and their preparations are official:

Solution of ammonium acetate containing about 7 per cent.....	$\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$.
Ammonium benzoate containing 98 per cent. absolute..	$\text{NH}_4\text{C}_6\text{H}_5\text{O}_2$.
Ammonium bromide containing 97 per cent. absolute..	NH_4Br .
Ammonium carbonate containing 97 per cent. of a mixture of.....	NH_4HCO_3 and $\text{NH}_4\text{NH}_2\text{CO}_2$.
Aromatic spirit of ammonia. A pharmaceutical.	
Ammonium chloride containing 99.5 per cent. absolute.....	NH_4Cl .
Troches of ammonium chlorid. A pharmaceutical.	
Ammonium iodide containing 97 per cent. absolute....	NH_4I .
Ammonium salicylate containing 98 per cent. absolute.....	$\text{NH}_4\text{C}_7\text{H}_5\text{O}_3$.
Ammonium valerate containing 98 per cent. absolute..	$\text{NH}_4\text{C}_5\text{H}_9\text{O}_2$.

LIQUOR AMMONII ACETATIS. Solution of Ammonium Acetate.

An aqueous solution which should contain not less than 7 per cent. of Ammonium Acetate [$\text{CH}_3\text{COONH}_4=76.51$], with small amounts of acetic and carbonic acids.

Recipe.—Ammonium Carbonate, five grammes. 5 Gm.
Diluted Acetic Acid, one hundred cubic centimeters. 100 Cc.

Add the Ammonium Carbonate (which should be in translucent pieces, free from white, pulverulent bicarbonate) gradually to the cold Diluted Acetic Acid, and stir until it is dissolved.

This preparation should be freshly made when wanted.

A clear colorless liquid, free from empyreuma, of a mildly saline, acidulous taste, and an acid reaction.

It is wholly volatilized by heat.

When Solution of Ammonium Acetate is heated with potassium hydroxide T.S., ammonia is evolved.

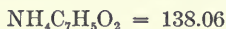
If to 5 Cc. of the Solution 1 Cc. each of sulphuric acid and alcohol be added, and the mixture boiled, the odor of acetic ether will be developed.

Remarks.—This solution, which contains about 7 per cent. of the salt, and which is known by the synonym of *spirit of mindererus*, is made by adding 5 Gm. of ammonium carbonate to 100 Cc. of diluted acetic acid, and the pharmacopœia directs that it should be freshly prepared when desired. The object of directing its being fresh is in order that the product will reach the patient in a slightly effervescent form, although the pharmacist must be careful not to cork the bottle while there is sufficient effervescence to produce an explosion. The pharmacopœia of 1880 directed a more convenient process of manufacture by ordering the ammonium carbonate to be dissolved in water and acetic acid be diluted with sufficient water, so that by mixing equal quantities of the two solutions a 7 per cent. solution of ammonium citrate will be produced. It seems unfortunate that this very convenient and simple process was dropped in the revision.

Solution of ammonium acetate is a refreshing refrigerant.

Dose.—16 Cc. (4 fluidrachms).

AMMONII BENZOAS. Ammonium Benzoate.



It should contain not less than 98 per cent. of pure Ammonium Benzoate [$\text{C}_6\text{H}_5\text{COONH}_4$] and should be kept in well-stoppered bottles.

Thin, white, laminar crystals, or a crystalline powder; odorless or having a slight odor of benzoic acid, a saline, bitter, afterward slightly acid taste, and gradually losing ammonia on exposure to the air.

Soluble in about 10.5 parts of water and 25 parts of alcohol at 25° C. (77° F.), in 1.2 parts of boiling water, and in 7.6 parts of boiling alcohol.

The salt fuses at 193° to 194° C. (379.4° to 381.2° F.), with decomposition, and when strongly heated emits vapors having the odor of ammonia and benzoic acid, and is finally completely volatilized.

The aqueous solution of the salt is neutral, or very slightly reddens blue litmus-paper.

Tests for Identity.—See Ammonium and Benzoates in Part V.

Impurities.—Sulphates, chloride, and heavy metals. Details in Part V.

Remarks.—This official is made by treating ammonia water with a molecular quantity of benzoic acid. There must, however, be a slight excess of ammonia, as the acid benzoate is less soluble than the official salt. It occurs in four-sided crystals, soluble in five parts of water, and is used as a diuretic.

Dose.—1 Gm. (15 grains).

AMMONII BROMIDUM. Ammonium Bromide.

$$\text{NH}_4\text{Br} = 97.29$$

It should contain not less than 97 per cent. of pure Ammonium Bromide and should be kept in well-stoppered bottles.

Colorless, transparent, prismatic crystals, or a white, crystalline powder; odorless, of a pungent, saline taste, and permanent in dry air.

Soluble in 1.2 part of water and in 12.5 parts of alcohol at 25° C. (77° F.), in 0.7 part of boiling water and in 9 parts of boiling alcohol.

When heated, Ammonium Bromide volatilizes completely, without fusing.

An aqueous solution of the salt slightly reddens blue litmus-paper.

Tests for Identity.—See Ammonium and Bromides in Part V.

Impurities.—Iodide, bromate, excess of iron, barium, heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made in several ways: first, by treating the ammonium sulphate with potassium bromide, and precipitating out the potassium sulphate thus formed, by the addition of alcohol; secondly, by treating ferrous bromide with ammonia water, and filtering the resulting ferric hydrate, and concentrating the filtered solution of ammonia bromide. Dr. Pile's process is by treating bromine with ammonia water, as shown in the following equation:



The bromine must be placed in a stone jar, with four times its weight of water, the ammonia gas being added very gradually to the mixture until the solution is free from the odor of bromine. Too much stress cannot be laid on the care required in adding ammonia water, as the rapid addition of ammonia may result in the formation of a very explosive nitrogen bromide (NBr_3).

Ammonium bromide is used as a hypnotic.

Dose.—1 Gm. (15 grains).

AMMONII CARBONAS. Ammonium Carbonate.

$$\text{C}_2\text{H}_{11}\text{N}_3\text{O}_5 = 156.01$$

It should contain not less than 97 per cent. of a mixture of Acid Ammonium Carbonate $[\text{CO}(\text{OH})\text{ONH}_2]$ and Ammonium Carbamate $[\text{CO}(\text{NH}_2)\text{ONH}_2]$, and should yield not less than 31.58 per cent. of ammonia gas. It should be kept in well-stoppered bottles, in a cool place. For dispensing purposes, only the translucent portions should be used.

White, hard, translucent, striated masses, having a strong odor of ammonia without empyreuma, and a sharp, saline taste. On exposure to the air, the salt loses both ammonia and carbon dioxide, becoming opaque, and is finally converted into friable, porous lumps, or a white powder.

Slowly but completely soluble in about 4 parts of water at 25° C. (77° F.); it is decomposed by hot water, with the elimination of carbon dioxide and ammonia. By prolonged boiling with water the salt is completely volatilized. Alcohol dissolves the carbamate and leaves the acid carbonate.

When heated, Ammonium Carbonate is completely volatilized, without charring.

The aqueous solution effervesces with acids, and shows an alkaline reaction with red litmus-paper.

If an aqueous solution containing 1 Gm. of the salt be slightly supersaturated with nitric acid, and then evaporated to dryness on a water-bath, it should afford a colorless and odorless residue, which, upon gentle ignition, should be completely volatilized (absence of *empyreumatic* or *non-volatile matters*).

Tests for Identity.—Given above.

Impurities.—Heavy metals, sulphate, thiosulphate, excess of chloride. Details in Part V. Empyreumatic matter, given above.

Assay.—Details in Part V.

Ammonium carbonate is not a true carbonate, the formula of which would be $(\text{NH}_4)_2\text{CO}_3$, analogous to Na_2CO_3 . The official salt is the combination of ammonium bicarbonate, $(\text{NH}_4)\text{HCO}_3$ (analogous to NaHCO_3), with ammonium carbamate. This carbamate is a complex substance, the derivation of which is here given as a matter of general interest.

Let us place side by side the graphic formulas of carbon dioxide, carbonic acid, and urea, a constituent of the urine, which has historic interest as the first organic substance that was made synthetically:

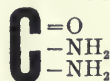
Carbon dioxide.
 CO_2



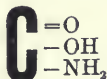
Carbonic acid.
 H_2CO_3



Urea.
 $\text{CO}(\text{NH}_2)_2$



Carbamic Acid is half-way between carbonic acid and urea, its graphic formula being—



or HNH_2CO_2 . Therefore, ammonium carbamate is $\text{NH}_4\text{NH}_2\text{CO}_2$.

This is the first carbonate we have discussed that deviates from the theoretic formula, such as can be developed by the method given on p. 377. We find, however, that there are several other official carbonates that are irregular as to formula, and among them may be cited the carbonates of magnesium, zinc, and bismuth.

The official ammonium carbonate can be converted into the true carbonate by dissolving in water containing ammonia.

If we take ammonium bicarbonate..... NH_4HCO_3
and add to it ammonia..... NH_3

we get neutral ammonium carbonate..... $(\text{NH}_4)_2\text{CO}_3$

Likewise, if we take ammonium carbamate... $\text{NH}_4\text{NH}_2\text{CO}_2$
and add to it water..... H_2O

we get neutral ammonium carbonate..... $\text{NH}_4\text{NH}_4\text{CO}_3$

or $(\text{NH}_4)_2\text{CO}_3$.

Official ammonium carbonate can also be considered as $(\text{NH}_3)_3(\text{CO}_2)_2, \text{H}_2\text{O}$.

Ammonium carbonate is prepared by subliming a mixture of crude ammonium sulphate with calcium carbonate. It is interesting to note that attempts to prepare same by aqueous crystallization have never met with success, hence attempts to restore effloresced ammonium carbonate by crystallization are futile.

Ammonium carbonate is a stimulant of particular value in diseases affecting the pulmonary mucous membrane, and hence it is used in pneumonia.

Dose.—250 milligrammes (4 grains).

Rot!

SPIRITUS AMMONIÆ AROMATICUS. Aromatic Spirit of Ammonia.

Recipe.—**Ammonium Carbonate**, in translucent pieces,
thirty-four grammes..... **34 Gm.**
Ammonia Water, *ninety cubic centimeters*..... **90 Cc.**
Oil of Lemon, *ten cubic centimeters*..... **10 Cc.**
Oil of Lavender Flowers, *one cubic centimeter*.... **1 Cc.**
Oil of Myristica, *one cubic centimeter*..... **1 Cc.**
Alcohol, *seven hundred cubic centimeters*..... **700 Cc.**
Distilled Water, *a sufficient quantity*,
 To make *one thousand cubic centimeters*.. **1000 Cc.**

To the Ammonia Water, contained in a flask, add *one hundred and forty cubic centimeters* of Distilled Water, and afterward the Ammonium Carbonate reduced to a moderately fine powder. Close the flask and agitate the contents until the Ammonium Carbonate is dissolved and allow it to stand for twelve hours. Introduce the Alcohol into a graduated bottle of suitable capacity, add first the Oils, then gradually the solution of Ammonium Carbonate, and afterward enough Distilled Water to make the product measure *one thousand cubic centimeters*. Set the liquid aside during twenty-four hours in a cool place, occasionally agitating, then filter it through paper, in a well-covered funnel.

Keep the product in glass-stoppered bottles, in a cool place.

A nearly colorless liquid when freshly prepared, but gradually acquiring a somewhat darker tint.

It has the pungent odor and taste of ammonia.

Specific gravity: about 0.900 at 25° C. (77° F.).

Remarks.—This is prepared by dissolving ammonium carbonate in ammonia water and the water; by dissolving the oils of lemon, lavender, and nutmeg in alcohol, and then mixing the two solutions.

In making this preparation ammonia water is added to the official ammonium carbonate, and this means adding ammonia to the bicarbonate and water to the carbonate; in other words, the solution of the official carbonate, in water containing ammonia, converts that mixed salt into the true carbonate, as shown above. This ammoniacal solution of the true carbonate is added to an alcoholic solution of the volatile oils of lemon, lavender flowers, and nutmeg. But the conversion of the bicarbonate and carbamate of the official salt into the true carbonate occurs completely only when the official salt is in "translucent pieces," as directed by the pharmacopœia. Translucent means intermediate between transparent and opaque. One cannot see through a translucent substance as one can through a pane of glass, but some light passes through the substance. Ground glass or thin pieces of horn are good examples of translucent substance. Examination of ammonium carbonate will show that it is hard to keep it in translucent pieces, the lumps become covered with white powder, which is wholly ammonium bicarbonate; that is, the carbonate gradually changes into the bicarbonate by the loss of ammonia and the absorption of moisture from the air. So eventually the entire mass of a sample of official carbonate will be converted into the bicarbonate, falling to a powder.

What happens when we try to use such effloresced carbonate—really bicarbonate—in the aromatic spirit? The ammonia water directed by the official process is only sufficient to convert the theoretic quantity of bicarbonate contained in the official salt into normal carbonate, while the balance of the bicarbonate, which would have been carbonate were the salt in translucent pieces, dissolves

unchanged. So far, good! but the evil effects come when we begin to add the aqueous solution to the alcoholic solution. The carbonate is soluble in alcohol, the bicarbonate is not, and if the aqueous solution contains any bicarbonate, the latter will be precipitated so soon as its solution is added to the alcoholic liquid.

Aromatic spirit of ammonia, when freshly prepared, is colorless, but on standing it gradually becomes dark, eventually turning to an amber tint. This darkening has been ascribed to two causes, first, that it is due to some chemical action between the oils which are used and the alkali, and, second, as a result of the reaction between the traces of tannin found in the alcohol which has been stored in barrels and traces of iron found in the ammonia. For the latter reason the pharmacopœia of 1880 directed the use, for this preparation, of alcohol that had been stored in glass vessels. Whatever the cause, the pharmacist should prepare his customer for any change in the color of the two samples of this or any other preparation when a prescription for the same is repeated. In this special case it is better to dispense an aromatic spirit of ammonia which has aged sufficiently to be somewhat colored. At any rate, a pharmacist should never let a customer obtain a preparation of a color different from that of the first sample without calling the customer's attention to the difference in coloration and giving the reason for same.

Dose.—2 Cc. (30 minims).

AMMONII CHLORIDUM. Ammonium Chloride.



It should contain not less than 99.5 per cent. of pure Ammonium Chloride.

A white, crystalline powder, without odor, having a cooling, saline taste, and permanent in the air.

Soluble in 2 parts of water, in 50 parts of alcohol, and in 5 parts of glycerin at 25° C. (77° F.), and in 1 part of boiling water.

On ignition, Ammonium Chloride is completely volatilized, without charring.

The aqueous solution of the salt (1 in 20) in ice-cold water should not redden blue litmus-paper at once; it affords, with silver nitrate T.S., a white, curdy precipitate, which is soluble in ammonia water.

The aqueous solution, when gently heated with potassium hydroxide T.S., evolves ammonia.

Tests for Identity.—Given above.

Impurities.—Heavy metals, sulphate, barium, calcium, sulphocyanate, excess of iron. Details in Part V. Emypreumatic and non-volatile matter, see Ammonium Carbonate.

Assay.—Details in Part V.

Remarks.—This salt is the oldest of the ammonium compounds, and, in fact, the word ammonia was derived from its synonym, *sal ammoniac*. Legend tells us *sal ammoniac* was first prepared in the Libyan desert near the temple of Jupiter Ammon, and for this reason it was called *sal ammoniac*.

Recent investigators, however, claim that the *sal ammoniac* of Herodotus and Pliny was nothing but common salt, and that the first reference to ammonium chloride is found in writings of Geber (eighth century), in the Latin translations of which book it is called *sal armoniac*.

Ammonium chloride is made by subliming ammonium sulphate with common salt. According as the apparatus is arranged to collect the vapors in a dome or in a vessel at one side we obtain the ammonium chloride in the form of a cake or in a fine powder. (See Sublimation, p. 107.) Since sublimation is usually carried on in iron vessels, commercial ammonium chloride frequently contains traces of iron. These can be removed by treating the solution with ammonia water and granulating the filtrate. At present a large amount of ammonium chloride is made by subliming a crude chloride, obtained by passing illuminating-gas through hydrochloric acid instead of sulphuric acid. Commercial sal ammoniac is in the form of white, translucent, tough cakes, about two inches thick, convex on one side and concave on the other, being formed as a crust in the dome of the subliming apparatus. The official ammonium chloride is a white, crystalline powder, permanent in the air, and soluble in three parts of water, but almost insoluble in alcohol. This crystalline powder is obtained usually by granulation.

The ammonium chloride, as just mentioned above, is quite volatile, and its vapors cause red litmus-paper to turn blue, and blue litmus-paper to turn red. Such substances as convert both colors of litmus-paper to the opposite colors are termed *amphoteric*. In the case of the sal ammoniac this phenomenon has been used as the basis of the claim that ammonium compounds are not definite compounds, but merely combinations of ammonia gas with the corresponding acid.

Ammonium chloride is a valuable stimulant and expectorant, and its vapor is frequently used as an inhalant in bronchial troubles, and numberless inhalers have been devised for its administration. In most cases their principle of action is the sucking into the nose or throat of air which has passed through hydrochloric acid and ammonia, and which is thus charged with ammonium chloride.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

AMMONII IODIDUM. Ammonium Iodide.

$\text{NH}_4\text{I} = 143.83$

It should contain not less than 97 per cent. of pure Ammonium Iodide and should be kept in small amber-colored, well-stoppered vials, protected from light.

When deeply colored, the salt should not be dispensed, but it may be deprived of free iodine by adding to its concentrated aqueous solution sufficient ammonium sulphide T.S. to render it colorless, then filtering, and evaporating on a water-bath to dryness.

Minute, colorless, cubical crystals, or a white, granular powder, without odor when colorless, but emitting a slight odor of iodine when colored, and having a sharp saline taste. The salt is very hygroscopic, and soon becomes yellow or yellowish-brown on exposure to the air and light, owing to the loss of ammonia and the liberation of iodine.

Soluble in 0.6 part of water and in 9 parts of alcohol at 25°C . (77°F .); in 0.43 part of boiling water and in 3.7 parts of boiling alcohol.

When heated on platinum foil, Ammonium Iodide evolves vapor of iodine, and volatilizes completely without fusing.

Tests for Identity.—See Ammonium and Iodides in Part V.

Impurities.—Barium, excess of iron, excess of free iodine, heavy metals, not more than 3 per cent. of bromide or chlorides. Details in Part V.

Remarks.—This official is made by treating the ammonium sulphate with potassium iodide, as shown in the following equation:



The resulting potassium sulphate is separated from the ammonium iodide by precipitation with alcohol. It might be well here to refer to the ingenuity exercised in attempts by the manufacturing chemist to remove the by-products from the chemical desired. It will be noticed that, whenever possible, the chemist arranges that the by-product be an insoluble salt when the product desired is soluble. This, for instance, is shown in the manufacture of the solution of soda (p. 1115). When the product desired is insoluble, then the manufacturer aims to make the by-product a soluble body. This is shown in the manufacture of calcium carbonate (p. 1121).

In one case the soluble body is freed from the insoluble body by filtration; in the other case, the insoluble substance is freed from the soluble side-product by washing. But as the formation of an insoluble side-product is sometimes difficult, as was shown in the case of the manufacture of hydrobromic acid, recourse was had, in the Fothergill process, to the sparing solubility of potassium bitartrate, and into this salt was the potassium bromide converted by use of tartaric acid. In hydrobromic acid we have a product which can be separated from the side-product by distillation, and in the commercial process distillation is employed, but in the case of the ammonium iodide none of the schemes of separation just described is applicable, it being impracticable to form an insoluble or sublimable potassium salt, so recourse is had to the solubility of the ammonium iodide in alcohol and the comparative insolubility of potassium sulphate in the same liquid. Hence the aqueous solution of the two salts is concentrated, alcohol is added, and the mixture cooled, and after the potassium sulphate has separated, the hydro-alcoholic solution of ammonium iodide is filtered and rapidly granulated by evaporation on a water-bath.

Ammonium iodide has the form of minute cubic crystals or white granular powder, very rapidly becoming yellowish or yellowish brown, due to the loss of ammonium and the separation of free iodine. So weak is the combination of the ammonium and the iodine that, during the process of evaporation, the iodine becomes liberated, and it is necessary to add ammonia water or, better, ammonium sulphide, from time to time.

Ammonium iodide is used as a resolvent. When dispensed for internal use, the pharmacist should be careful to see that the product is colorless, and if brown, to bleach with ammonium sulphide, as directed in the pharmacopœial definition given above.

Dose.—250 milligrammes (4 grains).

AMMONII NITRAS (U. S. P. 1890).

Ammonium nitrate was first made by Glauber (1667) by treating nitric acid with spirit of hartshorn.

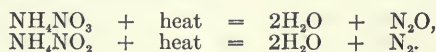
It is now usually made by treating ammonium carbonate with nitric acid and crystallizing by evaporation of the solution. It occurs in colorless crystals, soluble

in five parts of water, and, like all ammonium compounds, easily sublimed. It is interesting to note that when exposed to a current of dry ammonia gas its crystals absorb the gas and melt, yielding eventually a crystalline mass containing 25 per cent. of ammonia.

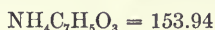
Its chief use is in the formation of *laughing-gas*, N_2O , which is yielded by heating the salt in a retort.

It is interesting to note that if, instead of the nitrate, ammonium nitrite is heated, nitrogen is produced.

Let us put the equations side by side:



AMMONII SALICYLAS. Ammonium Salicylate.



It should contain not less than 98 per cent. of pure Ammonium Salicylate $[C_7H_5(OH)COONH_4]$ and should be kept in well-stoppered bottles, protected from heat and light.

Colorless, lustrous, monoclinic prisms or plates, or a white, crystalline powder; odorless, and having at first a slightly saline, bitter taste, with a sweetish after-taste. Permanent in dry air.

Soluble in 0.9 part of water and 2.3 parts of alcohol at $25^\circ C.$ ($77^\circ F.$); freely soluble in boiling water and in 1 part of boiling alcohol.

When heated the salt fuses with decomposition, giving off inflammable vapors and an odor of phenol, and is finally completely volatilized.

The concentrated aqueous solution should be colorless, it should redden blue litmus-paper, and when gently heated with potassium hydroxide T.S., evolves ammonia.

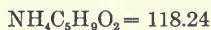
Tests for Identity.—See Ammonium and Salicylic Acid in Part V.

Impurities.—Heavy metals. Details in Part V.

Remarks.—This new official, made by neutralizing ammonia water with salicylic acid and either crystallizing or granulating the solution by evaporation on water-bath, is preferred by some physicians to sodium salicylate.

Dose.—250 milligrammes (4 grains).

AMMONII VALERAS. Ammonium Valerate.



[AMMONII VALERIANAS, PHARM. 1890]

It should contain not less than 98 per cent. of pure Ammonium Valerate $[C_5H_9COONH_4]$ and should be kept in well-stoppered bottles.

Colorless or white, quadrangular plates, emitting the odor of valeric acid, of a sharp and sweetish taste, and deliquescent in moist air.

Very soluble in water and in alcohol; also soluble in ether.

When heated the salt fuses, gives off vapor of ammonia and of valeric acid, and is finally completely volatilized.

Tests for Identity.—See Ammonium and Valerates in Part V.

Impurities.—Acetate, heavy metals. Details in Part V.

Remarks.—This preparation is made by passing dry ammonia gas into monohydrated valerianic (or valeric) acid. In the manufacture of this product it is very important to secure the proper valerianic acid. As noted on p. 688, most of the commercial acid is that containing three molecules of water, and this acid will not yield a satisfactory ammonium salt.

Ammonium valerate occurs in snow-white, four-sided, tabular crystals, having the characteristic odor of valeric acid, and a sharp, sweetish taste. It is used as a sedative in hysteria.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Ammonium Sulphate, $(\text{NH}_4)_2\text{SO}_4$.—Ammonium sulphate was recognized in the pharmacopœia of 1880, and deserves merely passing notice. As already mentioned, it is made in large quantity from ammoniacal liquor of gas-works, and is the commercial source of ammonia compounds. It can be made by combining ammonia water with sulphuric acid, as explained on p. 484.

Ammonium phosphate, $(\text{NH}_4)_2\text{HPO}_4$, was another official salt of the pharmacopœia of 1880. It is made by treating phosphoric acid with ammonia water. A modification of this, the double salt of ammonium and sodium phosphate, $\text{NH}_4\text{NaHPO}_4$, is used in chemical work under the name *microcosmic salt* or "*Phosphorsalz*." This salt, when melted on the loop of platinum, forms a transparent bead, similar to the borax bead, and when various metallic salts are melted therein, it assumes characteristic colors.

Another unofficial ammonium compound largely used in chemical work is the so-called *ammonium sulphide*. This is more properly called ammonium sulphhydrate, as the reagent used in precipitation in chemical work usually has the composition NH_4HS . However, the composition of this reagent varies, some samples containing a large excess of sulphur, and containing such bodies as $(\text{NH}_4)_2\text{S}_x$, $(\text{NH}_4)_2\text{S}_5$, and $(\text{NH}_4)_2\text{S}_7$.

This test solution is made by saturating ammonia water with hydrogen sulphide gas.

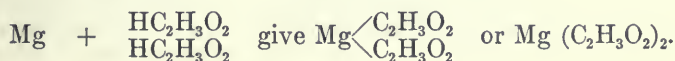
CHAPTER XXVIII

THE ALKALINE EARTH METALS

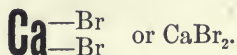
THE four elements, magnesium, calcium, strontium, and barium, are called alkaline earth metals because their oxides, MgO , CaO , SrO , and BaO , were called alkaline earths by early investigators—alkaline, because they turned red litmus blue, as do the alkalis, and earths, because differing from the alkalis in being found abundantly on the crust of the earth.

In passing from the alkalis—lithium, sodium, potassium, and ammonium—to the alkaline earths—magnesium, calcium, strontium, and barium—we not only pass into a new analytic group of metals, but also a group the valence of which is different.

The alkalis exhibit the valence 1; that is, one atom of an alkali metal replaces one atom hydrogen in an acid, or in combining directly with a negative element, takes up but one atom of that element's free bonds. The alkaline earths are bivalent—can replace two atoms of hydrogen in an acid. Thus, studying the formula of magnesium acetate, since one molecule of acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$, contains only one atom of replaceable hydrogen, two molecules of the acid will be required to furnish the two atoms of hydrogen demanded by one atom of magnesium, and the union will be as follows:



Again, bromine, having the valence I, and calcium, the valence II, unite as follows:



While sulphuric acid, H_2SO_4 , with two replaceable hydrogen atoms, combines with barium like this:



MAGNESIUM

Symbol, Mg. Atomic weight, approximately 24.

In the beginning of the eighteenth century a secret remedy was introduced under the name of "magnesia alba," thus differentiating it from the already popular *magnesia nigra*, which is now known as the ore *pyrolusite* (manganese dioxide) (p. 527). A recipe for this magnesia alba was published by Valentinus, of Giessen, in 1707, who obtained it by evaporation of the mother liquor from saltpeter, and lixiviation of the calcined residue.

Before this, magnesium sulphate was described by Nehemiah Grow, having been obtained by him by evaporation of the water of the Epsom spring.

Neither of these writers touched upon the composition of the two compounds, magnesium oxide and magnesium sulphate, and the chemical history of magnesium really begins with the investigation of Black (1755), who proved that magnesium carbonate was different from calcium carbonate or limestone. The metal was first isolated by Sir Humphry Davy in 1808, during his classic researches in electrolysis. In 1856 St. Clair Deville devised the chemical method for the isolation given below.

The chief ores of magnesium are the *magnesite*, just mentioned, which is magnesium carbonate; *dolomite*, a mixed carbonate of magnesium and calcium; *carnallite*, a mixture of potassium and magnesium chloride. (See p. 431.) Aside from these ores, from which the magnesium compounds used in pharmacy are generally derived, there are several magnesium minerals which are of interest other than pharmaceutic. This *serpentine*, a green stone, much used in building, is magnesium silicate, as is also *talcum* (*French chalk*), and *meerschaum*, well known as the substance of which the best pipes are made; *asbestos* is a mixed silicate of magnesium and calcium.

The chemical method of isolation of magnesium mentioned above is by heating magnesium chloride with metallic sodium, sodium chloride and calcium fluoride being added to the reacting mixture as a *flux*—that is, as an agent to remove contaminations in the form of a fusible mass. It is now made by the electrolysis of molten magnesium chloride in an atmosphere of hydrogen or nitrogen.

Magnesium is a light, silvery-gray metal, with the specific gravity of 1.75, and usually enters commerce in the form of rods or of thin ribbon. While affected by the air, it does not react with the same as

violently as do the alkaline metals. Hence it is unnecessary to keep it under petroleum, as was the case with sodium and potassium. When magnesium is brought into contact with a flame, it burns with a brilliant white light, with the formation of magnesium oxide. This property is made use of in the magnesium lantern for theatrical purposes, in which case the magnesium, in the form of ribbons, is allowed to burn at the point of contact of an oxyhydrogen flame. *Flash-light* consists of magnesium in finely powdered form, mixed with powdered potassium chlorate. This mixture, when blown into a flame, ignites instantaneously, producing a brilliant illumination, and is, therefore, largely used for photographic purposes. It should be borne in mind that flash-light is a substance by no means free from danger, and, in fact, should be handled with considerable caution. Several fatal accidents, due to the careless handling of flash-light, are on record, one of the most notable being the killing of three men in the laboratory of Wiley and Harris, in Philadelphia.

Among the tests for magnesium are: flocculent precipitate of magnesium hydroxide, produced when potassium hydroxide is added to a solution of a magnesium salt, and the white precipitate of magnesium carbonate, produced when the soluble carbonate is added to the solution of magnesium salt. The most characteristic test, however, is the granular precipitate of magnesium ammonium phosphate, produced when a solution of sodium phosphate and ammonia water is added to a solution of magnesium salt. In the manufacture of the salts of magnesium the carbonate is usually employed as a magnesium compound. The following compounds of magnesium and their preparations are official:

Magnesium oxide, 96 per cent. absolute MgO .

Heavy magnesium oxide, 96 per cent. absolute MgO .

Magnesium carbonate, approximately $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 + 5\text{H}_2\text{O}$, should contain not less than 38.4 per cent. MgO .

Solution of magnesium citrate. A pharmaceutical.

Magnesium sulphate, 99.7 per cent. absolute $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

Effervescent magnesium sulphate. Containing 50 per cent. crystalline MgSO_4 .

Talc. Purified talc. Hydrous magnesium silicate prepared from above.

To which might be added:

Ferric hydroxide with magnesium oxide, discussed among the iron compounds.

MAGNESII OXIDUM. Magnesium Oxide.

$\text{MgO} = 40.06$

[MAGNESIA, PHARM. 1890]

It should contain, after ignition, not less than 96 per cent. of pure Magnesium Oxide. It should be kept in well-closed vessels.

A white, very bulky, and very fine powder, without odor, and having an earthy, but not a saline taste. On exposure to the air, it slowly absorbs moisture and carbon dioxide.

Almost insoluble in water and insoluble in alcohol, but soluble in dilute acids.

When moistened with water it has a faintly alkaline reaction upon red litmus-paper.

On stirring 1 part of Magnesium Oxide with 15 parts of water, in a beaker, and allowing the mixture to stand for about half an hour, it will form a gelatinous mass of sufficient consistence to prevent it from dropping out when the beaker is inverted.

A solution of Magnesium Oxide in diluted sulphuric acid, mixed with ammonium chloride T.S. and an excess of ammonia water, yields, with sodium phosphate T.S., a white, crystalline precipitate.

If a mixture of 1 Gm. of Magnesium Oxide with 50 Cc. of water be heated to boiling, and after cooling, filtered, the filtrate should not show more than a faintly alkaline reaction with red litmus-paper, and when evaporated to dryness should not leave more than 0.005 Gm. of residue (limit of *foreign soluble salts*).

If Magnesium Oxide be exposed to a low red heat in a porcelain crucible, it should not lose more than 15 per cent. of its weight (limit of *water of hydration*).

Tests for Identity.—Given above.

Impurities.—Foreign soluble salts and excess of water of hydration given above. Excess of carbonate, excess of calcium, excess of iron, heavy metals. Details in Part V.

Assay.—Details in Part V.

MAGNESII OXIDUM PONDEROSUM. Heavy Magnesium Oxide.

MgO = 40.06

[MAGNESIA PONDEROSA, PHARM. 1890]

A white, dense, and very fine powder, which should conform to the reactions and tests given under *Magnesi Oxidum*.

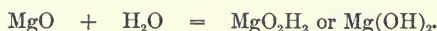
It differs, however, from the latter in not readily uniting with water to form a gelatinous hydroxide.

Remarks.—These two magnesiæ are prepared by the calcination of light and heavy magnesium carbonates respectively, and the carbonate can be made light or heavy as precipitated from cold dilute solutions or from hot concentrated solutions, as already explained in discussion of the formation of light and heavy precipitates (p. 142). Bear in mind that *magnesia is not magnesium*. Magnesium is the metal—the element; magnesia is the oxide of the metal, MgO, not the hydroxide, as was the case with soda and potassa.

The medicinal use of light and heavy magnesia is identical, both being laxative and antacid, the advantage of the heavy magnesia over the light magnesia depending entirely upon its greater density, the average heavy magnesia occupying only about one-quarter the bulk of the light magnesia. This should be remembered in considering the dosage of the two preparations, they being generally employed in domestic practice, and then given by measure rather than by weight. A dessertspoonful of light magnesia is generally given as the dose, while of the heavy magnesia, a half-teaspoonful to a teaspoonful is usually sufficient. In each case the customer should be directed to administer the substance in a moist condition. Thus, in giving the heavy magnesia, place from one to two teaspoonfuls of water in a tablespoon, and add thereto the teaspoonful of magnesia. The process should never be reversed, as the adding of water to the magnesia gives rise to hard lumps, whereas by the method just given a smooth mixture is obtained.

Under no circumstance should the pharmacist advise placing dry magnesia in the mouth, as the chemical will then adhere to the tongue and roof of the mouth, producing so disagreeable an impression that the victim will always remember the person giving such advice.

The addition of water to magnesia produces magnesium hydroxide by the following reaction:



Various combinations of magnesium hydroxide with water have been put on the market under the name of *milk of magnesia*. This is generally produced by the precipitation of a soluble magnesium salt with sodium hydroxide, and is scarcely more valuable than the hydroxide produced by treating the heavy magnesia with water.

Dose of both oxides of magnesium, as given by the pharmacopœia, is 2 Gm. (30 grains).

MAGNESII CARBONAS. Magnesium Carbonate.

Approximately $(\text{MgCO}_3)_4 \cdot \text{Mg}(\text{OH})_2 + 5\text{H}_2\text{O} = 482.26$.

Magnesium Carbonate $[(\text{CO}_3)_2\text{Mg}]_4 \cdot \text{Mg}(\text{OH})_2 + 5\text{H}_2\text{O}$ should yield, upon ignition, not less than 40 per cent. of residue, of which not less than 96 per cent. should consist of pure magnesium oxide.

Light, white, friable masses, or a bulky, white powder, without odor, and having a slightly earthy taste; permanent in the air.

Practically insoluble in water, to which, however, it imparts a slightly alkaline reaction; insoluble in alcohol, but soluble in dilute acids with effervescence.

When strongly heated, the salt loses water and carbon dioxide, and is converted into magnesium oxide.

If a mixture of 1 Gm. of Magnesium Carbonate with 50 Cc. of water be heated to boiling, and after cooling, filtered, the filtrate, if evaporated to dryness upon a water-bath, should leave not more than 0.001 Gm. of residue (limit of *foreign soluble salts*).

Tests for Identity.—See Magnesium and Carbonates in Part V.

Impurities.—Foreign soluble salts (see above). Excess of calcium, excess of iron, heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—It will be noticed that this chemical is one of the four official carbonates not possessing the formula of the true carbonate. (See p. 489.) In all these cases the deviation from the theoretic is not intentional, but is due simply to the fact that the true carbonates are unstable, and the formula given represents, as nearly as possible, the composition of the purest form which this chemical ordinarily assumes.

Magnesium carbonate is made by the double decomposition of magnesium sulphate with sodium carbonate.

According to conditions of chemical actions,—temperature and concentration of reacting solutions, treatment after formation, etc.,—the finished product can have most varying composition.

Thus, under differing conditions, from solutions of magnesium sulphate and sodium carbonate can be obtained $\text{MgCO}_3 \cdot 5\text{H}_2\text{O}$; $\text{MgCO}_3 \cdot 2\text{H}_2\text{O}$; $(\text{MgCO}_3)_2\text{Mg}(\text{OH})_2 \cdot 2\text{H}_2\text{O}$; $(\text{MgCO}_3)_3\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$; and $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2$, combined with five to eleven molecules of water respectively.

The usual commercial carbonate has the composition $(\text{MgCO}_3)_4\text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$, and this formula is assigned to it by the United States Pharmacopœia.

In other words, official magnesium carbonate is a combination of four molecules of true magnesium carbonate with one molecule of magnesium hydroxide and five molecules of water.

As mentioned above, according as the precipitation takes place in a cold diluted solution or in hot concentrated solutions, do we obtain light precipitated carbonate or heavy precipitated carbonate.

As far as the carbonate itself is concerned, this is of little importance, but when we consider that the two official forms of magnesia are derived from the light and heavy carbonates respectively, then the question forces itself upon our notice.

The official magnesium carbonate is a light form, the heavy carbonate being of interest merely in the formation of heavy magnesia, and for this purpose manufacturers resort to extraordinary pains to manufacture a dense magnesium carbonate.

Magnesium carbonate is a valuable antacid and a laxative.

Dose.—3 Gm. (45 grains).

LIQUOR MAGNESII CITRATIS. Solution of Magnesium Citrate.

Recipe.—Magnesium Carbonate, fifteen grammes. 15.0 Gm.
Citric Acid, thirty-three grammes. 33.0 Gm.
Syrup of Citric Acid, sixty cubic centimeters. 60.0 Cc.
Potassium Bicarbonate, two and one-half grammes 2.5 Gm.
Water, a sufficient quantity.

Dissolve the Citric Acid in *one hundred and twenty cubic centimeters* of Water, and, having added the Magnesium Carbonate, stir until it is dissolved. Filter the solution into a strong bottle of the capacity of about *three hundred and sixty cubic centimeters*, containing the Syrup of the Citric Acid. Then add enough Water to nearly fill the bottle, drop in the Potassium Bicarbonate, and immediately stopper the bottle securely. Lastly, shake the mixture occasionally, until the Potassium Bicarbonate is dissolved. This Solution should be freshly prepared when wanted.

Remarks.—This official solution is made by treating the carbonate with citric acid, sweetening and flavoring this with syrup of citric acid, and bottling with an excess of carbon dioxide formed by addition of potassium bicarbonate. The finished product, dispensed in stout bottles securely wired, is popular under the name of *purgative lemonade*.

This recipe has been criticized because the product does not keep. On standing a few weeks there forms within the tightly corked bottle stringy masses, which not only spoil the appearance, but also destroy the pleasant taste. These masses are none other than microscopic plants, produced by spores introduced into the bottle before filling, and thriving in the weak saccharine solution. The formation of these plants can be completely prevented by the simple precaution of sterilization. If care is taken to scald the bottles before putting in the solution, immersing corks in boiled water before being used, and using only freshly boiled water in preparing the solution and filtering through white paper, *not* the common gray, a product is obtained which, in the writer's experience, has kept perfectly for several months.

Occasionally, even with these precautions, a precipitation occurs in the bottled solution. In such cases, however, the precipitate is dense rather than stringy, and is due to use of an impure magnesium carbonate.

Dose.—360 Cc. (12 fluidounces).

Effervescent magnesium citrate (U. S. P. 1890) is prepared by treating magnesium carbonate with citric acid, with sufficient water to produce the chemical action. The resulting pasty mass is then dried, preferably on a radiator, and then reduced to a powder and mixed with sodium bicarbonate, more citric acid, and sugar. This mixture is then moistened with strong alcohol, and granulated by passing through a coarse sieve, as described on p. 149. The official product must be put in warm, com-

pletely dried bottles, which should be sufficiently tightly sealed to prevent access of air.

The effervescent citrate is a very agreeable preparation, for when it is added to water, a refreshing effervescent draught is readily obtained. It is usually administered in tablespoonful doses.

MAGNESII SULPHAS. Magnesium Sulphate.



It should contain not less than 99.7 per cent. of pure Magnesium Sulphate [$\text{SO}_4, \text{O}_2\text{Mg} + 7\text{H}_2\text{O}$] and should be kept in well-closed vessels.

Small, colorless, prismatic needles or rhombic prisms, without odor, and having a cooling, saline, and bitter taste; slowly efflorescent in the air.

Soluble in 0.85 part of water at 25° C. (77° F.) and in 0.13 part of boiling water; insoluble in alcohol.

When heated to 52° C. (125.6° F.) or exposed to warm air, the salt loses one molecule of water, and is converted into a white powder. At about 130° C. (266° F.) it still retains 1 molecule of water, and at a temperature between 200° and 238° C. (392° and 460.4° F.) it is rendered anhydrous.

An aqueous solution of Magnesium Sulphate is neutral to litmus-paper.

Tests for Identity.—See Magnesium and Sulphates in Part V.

Impurities.—Heavy metals and arsenic. Details in Part V.

In magnesium sulphate—the well-known *Epsom salt*—we have the sulphate of a metal with the valence II.

Taking sulphuric acid, H_2SO_4 , as the type, we find that, in each of the sulphates hitherto considered, the two hydrogen atoms in the acid were replaced by two atoms of the metal, since the four alkaline metals are univalent. On the other hand, the four alkaline earth metals, which we are now studying, have the valence II; that is, one atom of these metals can replace two atoms of hydrogen in an acid. The difference in these two groups of metals is best shown in a comparison of their sulphates, as here given.

Sulphates of the alkali metals:

K_2SO_4	Na_2SO_4	Li_2SO_4	$(\text{NH}_4)_2\text{SO}_4$
Potassium sulphate.	Sodium sulphate.	Lithium sulphate.	Ammonium sulphate.

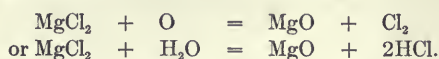
Sulphates of alkaline earth metals:

MgSO_4	CaSO_4	SrSO_4	BaSO_4
Magnesium sulphate.	Calcium sulphate.	Strontium sulphate.	Barium sulphate.

As above mentioned, magnesium sulphate contains seven molecules of water of crystallization. Of these, six molecules are lost by heating the crystals to 100° C., while the seventh molecule requires a heat of at least 200° C. to dry it off. This leads physicists to consider the linkage between the seventh molecule of water and the rest of the compound as stronger than is the case with the other six, and it has given rise to a physical distinction in water of crystallization, the comparatively easily removed water being called water of crystallization, while the molecule of water tenaciously holding to the molecule being called water of constitution. This topic has already been touched upon on p. 147.

Magnesium sulphate is made on an enormous scale commercially from the native magnesium minerals, dolomite or magnesite being generally employed. In the case of the first, the dolomite is calcined, whereby we obtain a mixture of the oxides of calcium, magnesium, and iron. The calcined mass is then treated with hydrochloric acid, producing the chlorides of the three metals. The filtered solution is

then evaporated, and during this process the magnesium chloride dissociates, as shown in the following equation:



Hence the evaporated mass is a mixture of the chlorides of calcium and of iron, while the magnesium has been converted into sparingly soluble magnesium oxide. The two chlorides just mentioned are readily removed from the magnesium by treatment with water, and the magnesia residue thus freed from the iron and calcium is then treated with sulphuric acid, and the resulting sulphate purified by crystallization. In view of the peculiarity just mentioned regarding the instability of magnesium chloride, it might be here stated that the one way of obtaining a dry magnesium chloride is by the evaporation of a mixed solution of magnesium chloride and ammonium chloride.

Magnesium sulphate is a valuable saline cathartic and is a constituent of many of the most popular purgative waters. The chief objection to its use is the disagreeably bitter taste which the salt possesses, and it might here be stated that this disagreeable taste can be much lessened by administering the solution in an ice-cold condition.

Dose.—16 Gm. (240 grains).

MAGNESII SULPHAS EFFERVESCENS. Effervescent Magnesium Sulphate.

<i>Recipe.</i> —Magnesium Sulphate, uneffloresced crystals, five hundred grammes.....	500 Gm.
Sodium Bicarbonate, dried and powdered, four hundred and three grammes.....	403 Gm.
Tartaric Acid, dried and powdered, two hundred and eleven grammes.....	211 Gm.
Citric Acid, uneffloresced crystals, one hundred and thirty-six grammes.....	136 Gm.
To make about one thousand grammes..	1000 Gm.

Dry the Magnesium Sulphate on a water-bath until it ceases to lose weight, then, after powdering the dry salt, mix it intimately with the Citric Acid, which has previously been powdered, and the Tartaric Acid, and thoroughly incorporate the Sodium Bicarbonate. Place the mixed powders on a plate of glass or in a suitable dish, in an oven heated to between 93° and 104° C. (199.4° and 219.2° F.).

When the mixture has acquired a moist consistence by the aid of careful manipulation with a wooden spatula, rub it through a No. 6 tinned-iron sieve, and dry the granules at a temperature not exceeding 54° C. (129.2° F.). Keep the product in well-stoppered bottles.

Remarks.—This new official, containing 50 per cent. of the crystalline salt, replaces the granular effervescent magnesium citrate of the old pharmacopœia. Whether the change is an improvement is questionable.

Dose.—16 Gm. (240 grains).

TALCUM. Talc.

A native hydrous magnesium silicate.

A white or grayish-white powder or grayish-green irregular masses of waxy lustre; when rubbed upon the skin it imparts a feeling like greasiness; permanent in the air. Inodorous and tasteless.

Insoluble in water and in dilute solutions of the acids and alkali hydroxides.

Specific gravity: 2.2 to 2.8.

Mix 0.5 Gm. of Talc with about 2 Gm. each of anhydrous sodium and potassium carbonates, heat the mixture in a platinum crucible until fusion is complete, and treat the resulting fused mass with hot water; then neutralize the liquid with diluted sulphuric acid, and, after adding an additional 10 Cc. of the latter, evaporate the mixture until the white fumes of sulphuric anhydride are evolved, then add 20 Cc. of water, and, after boiling the mixture and filtering, an insoluble residue of silica should be left. If to the filtrate ammonia water and ammonium chloride T.S. be added, a white, gelatinous precipitate of *aluminum hydroxide* may be formed, while the filtrate from this, upon the addition of sodium phosphate T.S., should yield a white, crystalline precipitate (*magnesium salts*).

If 1 Gm. of Talc be boiled with 25 Cc. of diluted hydrochloric acid for one-half hour, adding water from time to time to maintain approximately the original volume, and the liquid filtered, then the filtrate should yield, upon evaporating to dryness, igniting, and quickly weighing, not more than 0.05 Gm. of residue.

Tests for Identity.—Given above.

Remarks.—This new official, commonly called *soapstone*, or *French chalk*, was introduced because, when purified, it is an admirable filtering medium.

TALCUM PURIFICATUM. Purified Talc.

Recipe.—Talc, in fine powder, five hundred grammes. 500 Gm.
Hydrochloric Acid, seventy-five cubic centimeters. . . . 75 Cc.
Water, a sufficient quantity.

Mix the powdered Talc with about *twenty-five hundred cubic centimeters* of boiling Water, gradually add *fifty cubic centimeters* of the Hydrochloric Acid, and boil the mixture during fifteen minutes, then allow it to stand for fifteen minutes. Decant, and reject the supernatant liquid containing the finer particles of Talc in suspension, and again boil the residue with *twenty-five hundred cubic centimeters* of Water, mixed with the remainder of the Hydrochloric Acid, and allow it to stand for fifteen minutes. Again decant, and reject the finer suspended particles, and wash the coarser residue with Water by repeated decantation, until a portion of the wash-water, after filtering and acidifying with nitric acid, fails to become opalescent upon the addition of silver nitrate test solution. Then transfer the magma to a close linen or muslin strainer, allow it to drain, and dry it at 110° C. (230° F.).

Purified Talc, when subjected to ignition at red heat, should lose not more than 5 per cent. of its weight.

If 10 Gm. of Purified Talc be boiled with 50 Cc. of distilled water for one-half hour, adding water from time to time to maintain approximately the original volume, it should yield a filtrate which is neutral to litmus-paper, and one-half of this filtrate, when evaporated and dried at 110° C. (230° F.), should yield not more than 0.005 Gm. of residue (limit of *soluble substances*).

Impurities.—More than $\frac{5}{100}$ of 1 per cent. soluble matter. Details above. Iron. Details in Part V.

Remarks.—This is talc purified by treatment with hydrochloric acid. It is used as a filtering medium in those cases where precipitated calcium phosphate was directed by the pharmacopœia of 1890, such as in making aromatic waters and some of the syrups.

Magnesii Sulphis (U. S. P. 1890).—This former official is made by passing sulphurous acid gas through milk of magnesia (magnesium hydrate), as shown in the following equation:



Magnesium sulphite is used as an antiferment, and given in doses of from 1 to 2 Gm.

CALCIUM

Symbol, Ca. Atomic weight, approximately 40.

This element occurs as carbonate in the well-known minerals, *limestone*, *chalk*, and *marble*; *gypsum* (or in the exsiccated form, commercial *plaster of Paris*) is calcium sulphate, and the same substance in crystalline form constitutes the beautiful mineral, *alabaster*. *Fluorspar*, the transparent crystals used so much in optics, is a form of calcium fluoride.

The word calcium is derived from the Latin word *calx*, lime, which has been in use since ancient days, its manufacture from mussel-shells being described by Dioscorides, while the caustic effect of burnt lime is mentioned by Pliny. Berzelius and Pontin (1808) obtained calcium amalgam by electrolysis of lime in the presence of mercury, and Davy, in the same year, obtained the free metal in impure form by distilling the mercury from the amalgam.

The metal is now obtained by treating calcium with metallic sodium and zinc, the zinc aiding the sodium in removing the chlorine from the calcium chloride. It is also made by electrolysis.

Calcium is light yellow, ductile, malleable, quite stable in the air, but burning with a brilliant yellowish flame. In the so-called *calcium light*, lime or calcium oxide is employed, this substance burning with a brilliant white light when introduced into an oxyhydrogen flame.

Among the tests for calcium is the precipitate of calcium carbonate, produced when a soluble calcium salt is treated with a solution of an alkaline carbonate. More characteristic, however, is the white precipitate of calcium oxalate, produced in solutions of calcium salts on addition of ammonium oxalate solution. Turning to oxalic acid (p. 674), it will be found that lime is its antidote by reason of this very reaction, calcium compounds converting oxalic acid into the insoluble and harmless calcium oxalate.

In making most of the calcium compounds, the precipitated carbonate is employed.

The pharmacopœia recognizes the following compounds of calcium and their preparations:

Lime. At least 90 per cent. absolute CaO .

Solution of lime. At least $\frac{1}{100}$ of 1 per cent. absolute Ca(OH)_2 .

Lime liniment. A pharmaceutical.

Syrup of lime, containing about $6\frac{1}{2}$ per cent. lime.

Sulphurated lime. At least 60 per cent. CaS and also some CaSO_4 .

Calcium bromide. At least 97 per cent. absolute CaBr_2 .

Prepared chalk. Native CaCO_3 .

Compound chalk powder, containing 30 per cent. prepared chalk.

Chalk mixture, containing 20 per cent. compound chalk powder.

Precipitated calcium carbonate. At least 99 per cent. absolute CaCO_3 .

Calcium hypophosphite. At least 98 per cent. absolute $\text{Ca(PH}_2\text{O}_2)$.

Syrup of hypophosphites.

Compound syrup of hypophosphites. Pharmaceuticals.

Precipitated calcium phosphate. At least 99 per cent. absolute $\text{Ca}_3(\text{PO}_4)_2$.

Syrup of calcium lactophosphate. A pharmaceutical.

Exsiccated calcium sulphate. About 95 per cent. absolute CaSO_4 .

To which might be added:

Chlorinated lime. Considered among the chlorine compounds.

CALX. Lime.**CALCIUM OXIDE**

$$\text{CaO} = 55.68$$

Prepared by calcining white marble, or the purest varieties of native calcium carbonate, and containing, when in the anhydrous state, not less than 90 per cent. of pure Calcium Oxide. It should be kept in well-closed vessels, in a dry place.

Hard, white, or grayish-white masses, which, in contact with the air, gradually attract moisture and carbon dioxide, and fall to a white powder; odorless and having a caustic taste.

Soluble in about 760 parts of water at 25° C. (77° F.) and in about 1600 parts of boiling water; insoluble in alcohol. It forms readily soluble salts with diluted acetic, hydrochloric, or nitric acids.

When sprinkled with about half its weight of water, Calcium Oxide becomes heated and is gradually converted into a white powder (calcium hydroxide or slaked lime). When this is mixed with about 3 or 4 parts of water, it forms a smooth magma (milk of lime).

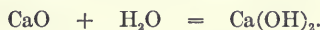
Its aqueous solution has an alkaline reaction upon red litmus-paper.

Tests for Identity.—Given above.

Impurities.—Excess of carbonate. Details in Part V.

Remarks.—This official represents the commercial product obtained by the calcination of limestone (p. 81). Chemically, it is more or less pure calcium oxide, and it might be said, in passing, that the lime made in northern Ohio usually contains strontium.

When treated with a small quantity of water, considerable heat is evolved, and the lump falls to a fine white powder. This is due to the formation of calcium hydrate by the chemical reaction between the water and the lime, which is expressed by the following equation:



The process is familiar under the name of *slaking lime*, the oxide being called *unslaked lime*, while the hydrate is slaked. In slaking small quantities of lime sometimes required in laboratory work, the heat is generated only in small quantities, so that at first glance it appears that no reaction is going on. The writer recalls one experience, when he rejected a sample of lime because of the difficulty of slaking, but found that, when he had thrown the same into water, a very energetic action would take place when a sufficient quantity was employed. This is particularly noticed in the slaking of small quantities required for making small amounts of solution of soda, and the writer's advice is for the operator to be guided by the eventual crumbling of the lump, instead of expecting an evolution of considerable heat.

Lime is used medicinally internally, when properly diluted with water, as an antacid. (See Lime Water.) When large quantities are applied, especially in the unslaked form, it acts as an escharotic and as a depilatory.

The use of lime in forming mortar in cements is so well known that the fact is here mentioned merely to call attention to that particular form called *Portland cement*. This is a mixture of unslaked lime, sand, and aluminum silicate, and is of value because, when immersed in water, it forms a hard mass resisting the action of water, due to the formation of calcium silicate. For masonry constructions under water it is invaluable.

LIQUOR CALCIS. Lime Water.**[SOLUTION OF CALCIUM HYDROXIDE]**

A saturated aqueous solution, which should contain not less than 0.14 per cent. of pure Calcium Hydroxide [$\text{Ca}(\text{OH})_2 = 73.56$].

The percentage of Calcium Hydroxide varies with the temperature at which the saturated solution is prepared, being about 0.17 per cent. at 15° C. (59° F.), the percentage diminishing as the temperature rises.

Recipe.—**Lime**, twelve grammes. **12 Gm.**
Distilled Water, a sufficient quantity.

Slake the Lime by the very gradual addition of *four hundred cubic centimeters* of Distilled Water, and agitate occasionally during half an hour. Allow the suspended particles to subside, decant the supernatant liquid, and reject it. Then add to the residue *thirty-six hundred cubic centimeters* of Distilled Water, agitate thoroughly, let the mixture stand for twenty-four hours, agitate again, then let the coarser particles of solid matter subside, and pour the liquid, holding the undissolved calcium hydroxide in suspension, into a glass-stoppered bottle. From time to time shake the bottle, so as to keep the solution saturated.

Pour off the clear liquid when required for use.

A clear, colorless liquid without odor, and having an alkaline taste.

It absorbs carbon dioxide from the air, a pellicle of calcium carbonate forming on the surface of the liquid.

On being heated it becomes turbid, due to the separation of calcium hydroxide, which redissolves when the liquid is cooled.

It gives a strongly alkaline reaction with red litmus-paper.

Tests for Identity. Given above.

Impurities.—Alkalis and alkaline carbonates. Details in Part V.

Assay.—Details in Part V.

Remarks.—This official is largely sold under the name of *lime water*, and contains about $\frac{1}{4}$ of 1 per cent. of calcium hydroxid. It represents a saturated solution of calcium hydroxide in water, and emphasis should be laid on the fact that a saturated solution is not necessarily a highly concentrated one. Another interesting point in connection with lime water is that the calcium hydroxide is more soluble in cold water than it is in hot water; and if a saturated solution prepared with cold water be warmed, it becomes turbid by the precipitation of the excess of lime.

Lime water is a preparation that is made by nearly all druggists, and that, unfortunately, in a careless way. Pharmacopœial lime water is *not* made by throwing a lump of lime into a crock, pouring water on it, and using the same lime for many subsequent crockfuls of water. In preparing lime water the pharmacopœial process should be used, and when once practised, it will be found to be of little less inconvenience than the crude method above described, and a highly superior product is obtained.

In preparing the solution of lime the pharmacopœia directs that the lime be slaked with a certain quantity of water, then mixed with water, and the lime allowed to precipitate; the water is then decanted and poured away. This is done because commercial lime usually contains more or less calcium chloride, which, if permitted to remain in the finished solution, would prove rather irritating. Calcium chloride is very soluble in water, and by macerating the lime with a small amount of water, the chloride will dissolve therein, and can be thus completely removed in the first water, which is thrown away.

The calcium hydroxide being sparingly soluble, this first water contains comparatively a small amount of lime, and in this way we rid ourselves of the calcium chloride with comparatively little loss of the calcium hydroxide. On throwing away the water containing the calcium chloride, the lime is then treated with a large quantity of water and the mixture allowed to stand. The pharmacopœia directs that, after shaking and waiting a long enough time for the coarser particles to subside, the liquid containing the undissolved lime in suspension is transferred to a glass bottle, and that this undissolved lime shall remain in the lime water until dispensed, in order that the solution be kept saturated. In practice, this part of the pharmacopœial requirement is rarely followed, and that in the interest of elegant pharmacy, as the pharmacopœia prohibits the filtration of the product, directing that the clear liquid be decanted when needed. Such procedure is almost sure to yield a more or less cloudy liquid, while most careful pharmacists desire to furnish a lime water which is as clear as crystal. In the writer's experience, half-gallon bottles containing the water with an excess of undissolved lime were kept on hand securely corked, and when the half-gallon dispensing bottle became empty, one of those kept in reserve was filtered, and a perfectly transparent liquid was obtained. The pharmacopœial objection to the use of filter-paper is based on the fact that the fibers of the filter absorb a certain quantity of calcium hydroxide, and, therefore, the first portion passing through the filter is deficient in lime strength. This disadvantage can be easily obviated by throwing away the first ounce or two of the filtered lime water and collecting the remainder in an appropriate container.

Solution of lime is a valuable antacid.

Dose.—16 Cc. (4 fluidrachms).

SYRUPUS CALCIS. Syrup of Lime.

This official is thoroughly discussed in Part VII., where the official recipe is given in full; so at this place it is only necessary to state that it is made by boiling together lime, sugar, and water.

It contains about 6.5 per cent. of calcium hydroxide. The greater solubility of lime in saccharine mixtures than in water alone is due to chemical action, the aldehyde-alcohol, sugar, combining with the lime to form what is commonly called calcium saccharate. (See p. 698.)

Dose.—2 Cc. (30 minims).

LINIMENTUM CALCIS. Lime Liniment.

This well-known official, *Carron oil*, has been mentioned on p. 272, while the chemistry of the preparation is referred to on p. 739. It suffices here to say that it is considered as a lime soap, and is a valuable remedy for burns and scalds. In fact, the name Carron oil is derived from the Carron Iron Works, where it was at first largely used for burns resulting from molten metal. In case of a severe burn it is applied freely on the injured surface, and it is then covered with pads of absorbent cotton.

CALX CHLORINATA. Chlorinated Lime.

This has already been considered on p. 391, under the preparations of chlorine, where it is explained that its entire value is due to chlorine content, the lime being merely the container for the valuable gas.

CALX SULPHURATA. Sulphurated Lime.

A mixture containing at least 60 per cent. of calcium sulphide [$\text{CaS} = 71.63$], together with unchanged calcium sulphate [$\text{CaSO}_4 = 135.15$], and carbon, in varying proportions.

Recipe.—**Exsiccated Calcium Sulphate**, in fine powder,
seventy grammes..... 70 Gm.
Charcoal, in fine powder, *ten grammes*..... 10 Gm.
Starch, *two grammes*..... 2 Gm.

Mix the powder thoroughly, pack the mixture lightly into a crucible, cover this loosely, and heat it to bright redness, until the contents have lost their black color. Allow the crucible to cool, reduce the product to powder, and at once transfer it to small, glass-stoppered vials.

A pale gray powder, exhaling a faint odor of hydrogen sulphide, having a nauseous and alkaline taste; gradually decomposing by exposure to moist air.

Very slightly soluble in cold water, more readily in boiling water, which partially decomposes it; insoluble in alcohol.

When Sulphurated Lime is decomposed by diluted acetic acid, calcium acetate is formed, hydrogen sulphide gas is evolved, and a residue of calcium sulphate and carbon remains.

The filtrate from this yields with ammonium oxalate T.S. a white precipitate, insoluble in acetic acid, but soluble in hydrochloric acid.

If 1 Gm. of Sulphurated Lime be added to a cold solution of 2.08 Gm. of cupric sulphate in 50 Cc. of water, followed by 10 Cc. of diluted hydrochloric acid, added in small portions, with constant stirring, and the mixture digested on a water-bath for 15 minutes and filtered, the addition of an excess of ammonia water should impart no color to the filtrate (presence of at least 60 per cent. of pure Calcium Sulphide).

Tests for Identity.—Given above.

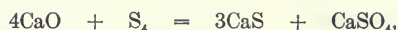
Assay.—Given above.

Remarks.—The common name of this chemical, "*Calcium Sulphide*," is incorrect, as it is not pure calcium sulphide, CaS , but a mixture of this with calcium sulphate and carbon. The process of manufacture in the present pharmacopœia is different from that of the pharmacopœia of 1880. The chemical is now prepared by the reduction of calcium sulphate with carbon, similar to the reduction process in the manufacture of sulphurous acid given on p. 425, only it is carried farther in the case of sulphurated lime, the reaction being—



However, part of the oxygen grasped by the carbon is taken from the air. Starch is also used in the present process, to furnish additional carbon as needed.

In the pharmacopœia of 1880 the chemical was made by heating a mixture of lime and sulphur. This yielded a similarly mixed product, for, as seen by the following reaction,



there is oxygen to be disposed of, thus giving rise to the sulphate.

Sulphurated lime is used as an alternative in skin diseases. It is indicated in that morbid condition of the system which manifests itself in that form of eruption called boils.

Dose.—65 milligrammes (1 grain).

The so-called calcium sulphide used as a depilatory—for the removal of superfluous hair—is, in truth, calcium sulphhydrate, that is, calcium sulphide combined with an extra molecule of hydrogen sulphide. This body is made by passing hydrogen sulphide gas through a thin paste of calcium hydroxide in water—which, by the way, is called *milk of lime*.

The equation of reaction is:



It will be noticed that the five preparations just discussed are considered as compounds of lime rather than as definite calcium salts, as those we are now ready to take up.

The true salts of calcium are usually prepared by treating the carbonate with the proper acid. Thus are made calcium bromide, CaBr_2 , and calcium chloride, CaCl_2 , the graphic formulas of which are good demonstrations of the fact that the calcium is a dyad:



CALCII BROMIDUM. Calcium Bromide.

$$\text{CaBr}_2 = 198.52$$

It should contain not less than 97 per cent. of pure Calcium Bromide and should be kept in well-stoppered bottles.

A white, granular salt, odorless, of a sharp, saline taste, and very deliquescent.

Soluble in 0.5 part of water and in 1 part of alcohol at 25° C. (77° F.); more soluble at boiling temperatures.

An aqueous solution of the salt is neutral to litmus-paper.

If 1 Gm. of Calcium Bromide be added to 20 Cc. of water, it should form a clear, colorless solution, leaving no residue (absence of *insoluble impurities*).

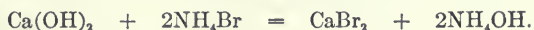
Tests for Identity. See Calcium and Bromides in Part V.

Impurities.—Iodides, heavy metals, barium, bromate, excess of nitrates and of ammonia. Details in Part V. Insoluble impurities given above.

This compound can be made by treating calcium carbonate with hydrobromic acid by the following equation:



or by the double decomposition of calcium hydroxide with ammonium bromide, as shown in the following equation:



In the former case the side product, carbon dioxide, passes off as gas, while in the second reaction the resulting ammonium hydroxide is dissipated during the evaporation of the solution of calcium bromide.

Calcium bromide is used as a hypnotic.

Dose.—1 Gm. (15 grains).

Calcium carbonate, CaCO_3 , is official in two forms.

CRETA PRÆPARATA. Prepared Chalk.

Native Calcium Carbonate, freed from most of its impurities by elutriation.

A white to grayish-white, very fine amorphous powder, often molded into conical drops; odorless and tasteless; permanent in the air.

Almost insoluble in water; insoluble in alcohol; soluble in diluted acetic, hydrochloric, or nitric acids, with copious effervescence, leaving not more than a trifling residue.

When heated to full redness, Prepared Chalk gradually loses carbon dioxide, and is converted into calcium oxide.

Tests for Identity.—Given above.

Remarks.—This represents the native ore, which is mined in large quantities in southwest England; freed from grit by elutriation, and the resulting magma then made into the characteristic cone—prepared chalk—by the process of trochiscation (p. 117).

A characteristic quality of prepared chalk is the stickiness which it possesses, and is preferable to the precipitated chalk in diarrhea mixtures for this very reason, being more likely to adhere to the irritated mucous membrane.

Prepared chalk is a constituent of *compound chalk powder* (p. 302), and this in turn is the main ingredient of *chalk mixture* (p. 264).

Dose.—1 Gm. (15 grains).

Whiting is an unofficial form of calcium carbonate, consisting of chalk less carefully washed than prepared chalk. School-crayons represent the same mineral shaped into sticks.

CALCII CARBONAS PRÆCIPITATUS. Precipitated Calcium Carbonate.

It should contain not less than 99 per cent. of pure Calcium Carbonate.

A fine, white powder, without odor or taste, and permanent in the air.

Nearly insoluble in water; the solubility is increased by the presence of ammonium salts, and especially by carbon dioxide; alkali hydroxides diminish its solubility; insoluble in alcohol; in diluted acetic, hydrochloric, or nitric acid it is completely soluble, with effervescence.

When heated to full redness, with access of air, the salt gradually loses carbon dioxide, and a residue of calcium oxide remains.

For applying tests of identity and of purity, 5 Gm. of Calcium Carbonate are mixed with 100 Cc. of distilled water, followed by hydrochloric acid, added drop by drop, with agitation, until solution takes place. The resulting solution should, after boiling and cooling, be of acid reaction, and there should not remain more than traces of insoluble matter.

In a portion of this acid solution, after neutralizing with ammonia water, ammonium oxalate T.S. produces a white precipitate of calcium oxalate, insoluble in acetic acid, but soluble in hydrochloric acid.

If to 20 Cc. of the acid solution ammonia water be added until of alkaline reaction, no turbidity or precipitation should take place either before or after boiling (absence of iron, aluminum, phosphates, etc.).

The acid solution should not respond to the Time-limit Test for heavy metals.

If 1 Gm. of the salt be agitated with 50 Cc. of water, the filtrate should not show an alkaline reaction with litmus-paper, and, on evaporation, should not leave a weighable residue (limit of soluble impurities).

Tests for Identity.—See Calcium and Carbonates in Part V.

Impurities.—Iron, aluminum, phosphates, excess of soluble impurities. See above. Heavy metals. Details in Part V.

Remarks.—*Precipitated chalk* is made from prepared chalk by dissolving the same in hydrochloric acid, whereby calcium chloride is produced, and then precipitated by the addition of sodium carbonate to the filtered solution of calcium chloride. The process is enlarged upon on p. 1120. It will be noted that the same precipitated chalk is the side-product in the manufacture of the three solutions already described—solution of soda, solution of potassa, and solution of chlorinated soda. The precipitated chalk so obtained, however, is scarcely fit for use, that from the first two products being largely contaminated with alkali, while that from Labarraque's solution smells strongly of chlorine.

The chief use of precipitated chalk is as a tooth-powder, and it is admirably adapted for the purpose in such preparations made by the retail pharmacist. Even better, however, is that form of calcium carbonate obtained by rubbing the soft interior of the cuttlefish bone against a sieve. As already mentioned, precipitated chalk is not as valuable as prepared chalk for diarrhea mixtures.

Dose.—1 Gm. (15 grains).

CALCII CHLORIDUM. Calcium Chloride.

$$\text{CaCl}_2 = 110.16$$

Calcium Chloride, rendered anhydrous by fusion at the lowest possible temperature. It should contain not less than 99 per cent. of pure Calcium Chloride and should be kept in well-stoppered bottles.

White, slightly translucent, hard fragments, odorless, having a sharp, saline taste.

It is very deliquescent.

Soluble in 1.3 parts of water and in 8 parts of alcohol at 25° C. (77° F.); in 1.5 parts of boiling alcohol and very freely soluble in boiling water, usually leaving a slight residue.

Below a red heat the salt fuses, and, on cooling, solidifies without change in composition.

When perfectly pure, Calcium Chloride dissolves in water without residue; the solution should be strictly neutral to litmus-paper.

When the salt has been overheated in fusing, the solution has an alkaline reaction, and a small residue of calcium oxide is left, which is soluble in hydrochloric acid.

If to the aqueous solution of the salt (1 in 20) ammonia water be added, until of alkaline reaction, no turbidity or precipitation should take place either before or after boiling (absence of *iron, aluminum, phosphates, etc.*).

If from 10 Cc. of the solution the calcium be completely precipitated by ammonium oxalate T.S., the filtrate should, on evaporation and ignition, leave not more than a trace of fixed residue (limit of *magnesium and alkalis*).

Tests for Identity.—See Calcium and Chlorides in Part V.

Impurities.—Iron, aluminum, phosphates, excess of magnesium or of alkalis, see above. Arsenic and lead. Details in Part V.

This substance, as already mentioned, is prepared by the treatment of calcium carbonate with hydrochloric acid, as shown in the following equation:

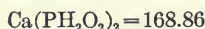


The solution is evaporated to dryness, and the product comes into the market either in the form of a white, granular powder, or in fused lumps.

Calcium chloride is official because of its value as a desiccating agent, as an absorber of water, it being used very largely in chemical work for this purpose. Thus, in keeping the air in the case of an analytic balance free from moisture, calcium chloride is employed. It is also used as a base for freezing mixtures, for, when combined with snow, it produces the exceedingly low temperature of -48°C .

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

CALCII HYPOPHOSPHIS. Calcium Hypophosphite.



It should contain not less than 98 per cent. of pure Calcium Hypophosphite $[(\text{PO}.\text{OH})_2.\text{Ca}]$ and should be kept in well-stoppered bottles; caution should be observed in dispensing Calcium Hypophosphite, as explosion is liable to occur when it is triturated or heated with nitrates, chlorates, or other oxidizing agents.

Colorless, transparent, monoclinic prisms, or small, lustrous scales, or a white, crystalline powder, odorless, having a nauseous and a bitter taste; permanent in the air.

Soluble in 6.5 parts of water at 25°C . (77°F .) and in 6 parts of boiling water; insoluble in alcohol.

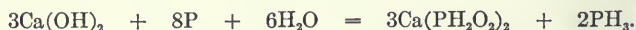
When heated in a test-tube the salt decrepitates, and above 300°C . (572°F .) it begins to decompose, giving off water and emitting inflammable gases (hydrogen and hydrogen phosphide), and leaving a residue of calcium pyrophosphate and metaphosphate, with some red phosphorus.

If 1 Gm. of the salt be added to 20 Cc. of water and well shaken, not more than a trace of residue should remain (absence of *phosphate* and *sulphate*).

Tests for Identity.—See Calcium and Hypophosphites in Part V.

Impurities.—Phosphate and sulphate, given above. Arsenic and heavy metals. Details in Part V.

This chemical is made by warming a mixture of phosphorus (finely divided, as explained on p. 117) and milk of lime at 40°C ., the reaction being



The solution of calcium hypophosphite is filtered from the excess of lime and is evaporated at low temperatures until a granular mass results.

The method of manufacture just mentioned is by no means free from danger, as mentioned when discussing the hypophosphites of potassium and sodium. Not only is the spontaneously inflammable *phosphine* (PH_3) evolved during process of manufacture, but also while granulating the salt. Hence the advice not to heat hypophosphites above 85°C . even on the water-bath.

In this connection it might be well to mention that it frequently happens that the calcium hypophosphite of commerce does not completely dissolve in the amount of water and acid directed for the manufacture of the official syrup of hypophosphites. This is due to the fact that such calcium hypophosphite is not up to the pharmacopœial requirements as to purity, and it is, therefore, good pharmacy always to choose a very reliable make of this salt for hypophosphite preparations. As is the case with all hypophosphites, this salt is supposed to be of value as a nutritive in phthisis. A constituent of

syrup of hypophosphites, U. S. P. (p. 195); of *compound syrup of hypophosphites*, U. S. P. (p. 195), and of *emulsion of cod liver oil with hypophosphites*, U. S. P. (p. 271).

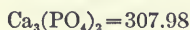
Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

SYRUPUS HYPOPHOSPHITUM. Syrup of Hypophosphites.

SYRUPUS HYPOPHOSPHITUM COMPOSITUS. Compound Syrup of Hypophosphites.

While these preparations are mentioned here because calcium hypophosphite is their chief constituent, the full explanation of them will be found on p. 195.

CALCII PHOSPHAS PRÆCIPITATUS. Precipitated Calcium Phosphate.



It should contain not less than 99 per cent. of pure Calcium Phosphate $[(\text{PO}_4)_2\text{Ca}]$.

A bulky, white, amorphous powder, odorless and tasteless; permanent in the air.

Almost insoluble in cold water; partly decomposed by boiling water, which dissolves out the acid salt; almost insoluble in acetic acid, except when freshly precipitated; easily soluble in hydrochloric or nitric acid; insoluble in alcohol.

At an intense white heat the salt fuses without decomposition.

When moistened with silver nitrate T.S., either before or after ignition, the salt acquires a yellow color (distinction from *acid calcium phosphate*, which, after ignition, when moistened with silver nitrate T.S., remains white).

For applying tests of identity and of purity, shake 2 Gm. of Precipitated Calcium Phosphate with 20 Cc. of water, add nitric acid, drop by drop, until solution is effected, and then add sufficient water to make the liquid measure 40 Cc. While making this solution no effervescence should occur on adding the acid (absence of carbonate).

Tests for Identity.—See Calcium and Phosphates in Part V.

Impurities.—Acid calcium phosphate, carbonate, given above. Excess of chloride, barium, heavy metals, and arsenic. Details in Part V.

Remarks.—*Precipitated calcium phosphate*, $\text{Ca}_3(\text{PO}_4)_2$, is obtained from bone-ash, 80 to 85 per cent. of which consists of this chemical.

In preparing it, bones are treated with hydrochloric acid, whereby soluble acid phosphate and calcium chloride are produced, as shown in the following equation:



and, on addition of ammonia water to the solution, the phosphate is again formed and precipitates out—



Bone-ash contains some magnesium salts, and the calcium phosphate obtained from this source is apt to contain ammoniomagnesium phosphate. For this reason much of the better grade of calcium phosphate is made by the action of sodium phosphate on calcium chloride

in the presence of ammonia. If the reaction takes place in neutral solution, hydrogen calcium phosphate $[\text{CaH}_4(\text{PO}_4)_2]$ is produced.

Because precipitated calcium phosphate is supposed to be practically insoluble in water, this salt was directed by the pharmacopœia of 1890 as the diluent and divider in the making of such preparations as aromatic waters and elixirs and tincture of opium. That the salt is completely insoluble when used for this process is an error, and frequently leads to mischievous results. Waters made with the precipitated phosphate rapidly become stringy, due to microscopic plants, while the alkaloids are rendered partly insoluble, with corresponding decrease in strength of the preparation. For this reason, at the revision of 1900, purified talc was chosen for aromatic waters instead of the phosphate.

Medical Properties.—Occasionally used as a tonic.

Dose.—1 Gm. (15 grains).

SYRUPUS CALCII LACTOPHOSPHAS. Syrup of Calcium Lactophosphate.

Of this official preparation, which contains 3 per cent. of calcium lactophosphas, full details are on p. 679, so here it suffices to say that the pharmacopœia directs its manufacture by treating calcium carbonate with lactic and phosphoric acids, along with sugar and flavor, and is given in doses of from 4 to 8 Cc.

CALCII SULPHAS EXSICCATUS. Exsiccated Calcium Sulphate.

A powder containing about 95 per cent., by weight, of Calcium Sulphate $[\text{CaSO}_4 = 135.15]$ and about 5 per cent. of water; prepared from the purer varieties of native gypsum $[\text{CaSO}_4 + 2\text{H}_2\text{O} = 170.91]$ by carefully heating until about three-fourths of the water has been expelled.

Exsiccated Calcium Sulphate should be kept in well-closed vessels, carefully protected from moisture.

A fine, white powder, without odor or taste.

From moist air it attracts water, becomes granular, and then loses the property of hardening with water.

When mixed with half its weight of water, Exsiccated Calcium Sulphate forms a smooth, cohesive paste, which rapidly hardens.

It is soluble in about 378 parts of water at 25°C . (77°F .) and in 451 parts at 100°C . (212°F .). In alcohol it is insoluble. It readily dissolves in diluted nitric or hydrochloric acid; also in saturated solutions of potassium nitrate, sodium thiosulphate, and of various ammonium salts.

When heated above 204°C . (399.2°F .) Exsiccated Calcium Sulphate becomes anhydrous and loses the property of forming a paste with water which hardens rapidly.

Tests for Identity.—See Calcium and Sulphates in Part V.

Impurities.—Carbonate. Details in Part V.

Remarks.—This official represents the native mineral gypsum in exsiccated form, and is commercially known as *plaster of Paris*. Its use in medicine and pharmacy is purely mechanical, and is due to the fact that when combined with water, it forms a paste which quickly settles to a firm, insoluble mass. This makes plaster of Paris of great value in the setting of broken limbs, while its pharmaceutic use in making fast the handles of pestles has already been commented on on p. 114.

STRONTIUM

Symbol, Sr. Atomic weight, approximately 87.

This element occurs in the minerals *strontianite* (strontium carbonate) and *celestite* (strontium sulphate).

The sulphate is found in considerable quantity in the group of islands in Lake Erie, especially at Put-in-Bay Island.

The carbonate was discovered near Strontian, a village of Scotland, and on being proved different from barium carbonate, was named by Hope, who proved that it contained a new metal by preparing several salts.

The metal was first isolated by Davy in 1808 by electrolysis of the oxide.

Strontium is now made by the electrolysis of strontium chloride.

Strontium is a brassy metal, having the specific gravity of 2.5, and comparatively stable in air. When placed in water, however, it decomposes same in a manner similar to that already explained under Potassium.

The tests for strontium and its compounds are, first, the crimson color which it transmits to a colorless flame, and, secondly, the sparingly soluble sulphate which is formed on adding a soluble alkaline sulphate to a soluble salt of strontium.

Strontium has comparatively little value in pharmacy, its chief use being in pyrotechnics. The red flame just described makes the strontium compounds valuable in this direction. Strontium is also used in the sugar industry for the regeneration of molasses.

The pharmacopœia recognizes three salts of strontium,—the bromide, the iodide, and the lactate,—the popularity of these leading to a demand for their admission into the United States Pharmacopœia of 1890, being chiefly developed through the advertising efforts of certain firms exploiting strontium compounds in medicine. The unprejudiced observer finds, however, scarcely sufficient value in strontium compounds to justify official recognition.

STRONTII BROMIDUM. Strontium Bromide.



It should contain not less than 97 per cent. of pure Strontium Bromide and should be kept in glass-stoppered bottles.

Colorless, transparent, hexagonal crystals; odorless and having a bitter, saline taste. Very deliquescent.

Soluble in about 1 part of water at 25° C. (77° F.) and in 0.4 part of boiling water. It is readily soluble in alcohol and is precipitated from this solution upon the addition of an equal volume of ether, in which it is insoluble.

When heated, the crystals at first melt and then lose all their water (30.4 per cent.). The anhydrous salt fuses at 630° C. (1166° F.).

To a non-luminous flame the salt communicates an intense red color.

Its aqueous solution is neutral to litmus-paper.

With calcium sulphate T.S. the aqueous solution (1 in 20) slowly forms a white precipitate of strontium sulphate, insoluble in dilute acids; the same reaction occurs more quickly with diluted sulphuric acid and the readily soluble sulphates.

With potassium chromate T.S. it forms a yellow precipitate of strontium chromate, soluble in acetic acid.

With ammonium carbonate T.S., or sodium carbonate T.S., it forms a white precipitate of strontium carbonate, soluble, with effervescence, in acetic acid.

Tests for Identity.—See above.

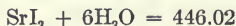
Impurities.—Iodide, heavy metals, and barium. Details in Part V.

Assay.—Details in Part V.

Remarks.—This compound is prepared by treating the carbonate with hydrobromic acid. It is used as a hypnotic.

Dose.—1 Gm. (15 grains).

STRONTII IODIDUM. Strontium Iodide.



It should contain not less than 98 per cent. of pure Strontium Iodide and should be kept in small glass-stoppered vials, carefully protected from light.

Colorless, transparent, hexagonal plates; odorless and having a bitter, saline taste. Deliquescent and colored yellow by exposure to air and light.

Soluble in about 0.5 part of water at 25° C. (77° F.) and in 0.27 part of boiling water. Also soluble in alcohol and slightly soluble in ether.

When cautiously heated, the crystals melt and gradually lose their water (24.05 per cent.), becoming anhydrous. At a red heat the salt is decomposed, losing iodine, and leaving a residue of strontium oxide.

To a non-luminous flame it imparts an intense red color.

Its aqueous solution is neutral or very slightly alkaline to litmus-paper.

Tests for Identity.—See Strontium and Iodides in Part V.

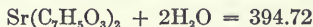
Impurities.—Heavy metals, barium. Details in Part V.

Assay.—Details in Part V.

Remarks.—This is made by treating strontium carbonate with hydriodic acid. It is used as an alterative.

Dose.—500 milligrammes (7½ grains).

STRONTII SALICYLAS. Strontium Salicylate.



It should contain not less than 98.5 per cent. of pure Strontium Salicylate $[(\text{C}_6\text{H}_4.\text{OH}.\text{COO})_2\text{Sr} + 2\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles, protected from heat and light.

A white, crystalline powder; odorless and having a sweetish, saline taste.

Soluble in 18 parts of water and in 66 parts of alcohol at 25° C. (77° F.); in 3.5 parts of boiling water and in 10.5 parts of boiling alcohol.

When heated the salt is decomposed, giving off inflammable vapors and an odor of phenol, and finally leaving a gray residue of strontium carbonate.

To a non-luminous flame its solution imparts an intense red color.

Its aqueous solution should be colorless and slightly alkaline to litmus-paper.

Tests for Identity.—See Strontium and Salicylates in Part V.

Impurities.—Heavy metals, excess of barium.

Assay.—Details in Part V.

Remarks.—This body is made by treating strontium carbonate with salicylic acid.

It is used in rheumatism.

Dose.—1 Gm. (15 grains).

BARIUM

Symbol, Ba. Atomic weight, 136.4

The chief ores of this metal are *heavy spar* (barium sulphate) and *witherite* (barium carbonate). The former ore was called "barote" by Guyton de Morveau (1779), from the Greek word *baros* (heavy), and from this the name of the element was derived.

An amalgam of barium and mercury was first obtained by Berzelius and Pontin in 1808, by electrolysis, but these investigators were unable to isolate the element, this being done by Davy the same year by distilling the mercury from the amalgam.

Barium is now obtained by electrolysis of the fused chloride.

Metallic barium resembles in many ways metallic strontium. It has a specific gravity of 3.75, is a brassy metal, and, like strontium, while stable in air, reacts with water, forming hydrogen and the hydrate of the metal.

The characteristic test for soluble barium compounds is the white precipitate formed by the addition of soluble sulphate. The barium sulphate thus produced is insoluble even in boiling nitric and hydrochloric acid, and is one of the most insoluble and infusible substances known. Barium compounds give to a colorless flame a green tint.

No salts of barium are official, but the pharmacopeia of 1890 recognized *barii dioxidum*, BaO_2 . This chemical is made by heating barium oxide, BaO , in contact with air. It was made official for the sole purpose of making hydrogen dioxide water by the recipe official in the United States Pharmacopœia of 1890.

The pharmacopeia also mentions in the Appendix two salts of barium used for testing purposes—barium chloride and barium nitrate—recipes for test solutions of each being given. The purpose of both of these solutions is for the detection of sulphates, they affording the most delicate test for that purpose.

CHAPTER XXIX

ZINC, MANGANESE, COBALT, AND NICKEL

REFERRING to the chapter on Analytic Chemistry (p. 985), it will be seen that the alkaline metals, lithium, potassium, sodium, and the radicle ammonium, form the first of the seven groups into which metals are divided in analysis. The alkaline earth metals, magnesium, calcium, strontium, and barium, form the second analytic group—those precipitated from solution by ammonium carbonate and ammonium phosphate.

The third analytic group includes the metals zinc, manganese, cobalt, and nickel; these four elements, along with aluminum, iron, and chromium, being precipitated from alkaline solution by the ammonium sulphide test solution.

The last three metals form the fourth analytic group, the third and fourth groups being differentiated by the fact that the fourth group can be precipitated from acid solution by the addition of ammonia water, while the third group is not precipitated by that reagent.

With the metals considered in this chapter we reach what might be called the commercial metals—that is, those which, by reason of stability, abundance, and general utility, are used largely in the arts as metals. The isolation of these metals from their ores has been practised for many centuries, and the process of extraction now in vogue is largely a modification of ancient methods. The study of the various methods of isolation forms the art called *metallurgy*.

ZINC

Symbol, Zn. Atomic weight, approximately 65.

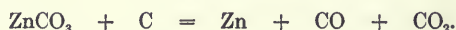
Zinc occurs as *calamine* (zinc carbonate) and *zinc blend* (zinc sulphide). Calamine is largely mined in the neighborhood of Aix-la-

Chapelle, Germany, and it is interesting that in that neighborhood there grows a wild violet which is characterized by the large amount of zinc in its ash, and it is, therefore, given the botanic name of *Viola calaminaris*. In this country zinc is mined in the neighborhood of Joplin, Mo., the ore found there being *sphalerite* (a form of zinc sulphide).

Zinc was early used as a constituent of brassy alloys, the coins of the Roman emperors, Nero and Hadrian, containing it.

For this purpose an ore of zinc was evidently used, Dioscorides and Pliny calling an ore that would turn copper yellow *cadmia*. Ore for similar purposes is called by Arabian writers *climia*, and from this word come such modifications as *calimia* and *lapis calaminaris*. The word zinc is first found in the writings of Basil Valentine and of Paracelsus, but the metal does not seem to have been isolated in Europe until Henkel produced it by a secret process, although Labavius (1597) describes the metal as a peculiar tin from East Indies.

Zinc is made by roasting calamine with charcoal and distilling the resulting zinc. Since calamine is a carbonate; extreme heat will calcine it, with the formation of zinc oxide. If charcoal be present during the calcination, it will act as a reducing agent, removing the oxygen in the form of carbon monoxide and carbon dioxide, by the following reaction:



This leaves metallic zinc in a more or less impure form, and the metal is, therefore, purified by continuing the heat until the zinc distills over.

If zinc sulphide is the ore employed, it is first roasted in air, when it decomposes into zinc oxide and sulphur dioxide, the latter passing off into the air or being collected in a Gay Lussac tower. The remaining zinc oxide is treated with charcoal, as in the case of calamine.

In technic there are several processes—English, Belgian, and Silesian—these differing only in the apparatus wherein occurs the reduction of the oxide with charcoal.

Zinc is the first metal which we have considered that is given official recognition; the three metals recognized by the pharmacopœia as uncombined metals being zinc, iron, and mercury.

ZINCUM. Zinc.

Zn = 64.9

It should contain not less than 99 per cent. of pure metallic Zinc.

A bluish-white metal, showing a crystalline fracture; in the form of thin sheets, or in irregular, granulated pieces, or molded into thin pencils, or in fine powder, and having a specific gravity ranging from 6.9 when it is cast to 7.2 after it is rolled.

Soluble in diluted sulphuric or hydrochloric acid with evolution of hydrogen.

When heated above 100° C. (212° F.) and not above 150° C. (302° F.), the metal becomes malleable and ductile; above 200° C. (392° F.) it becomes sufficiently brittle to be powdered in an iron mortar; at 412° to 415° C. (773.6° to 779° F.) it melts, and at 940° C. (1724° F.) it boils and may be readily distilled.

If 1 Gm. of Zinc be added to 20 Cc. of diluted hydrochloric acid, the liberated hydrogen should not have a disagreeable odor, nor should it color a strip of paper moistened with lead acetate T.S. (absence of *sulphur*) or with silver nitrate T.S. (absence of *arsenic*, *antimony*, and *phosphorus*). The resulting solution should be

clear and colorless, and should yield a white gelatinous precipitate with potassium ferrocyanide T.S., and a white precipitate with ammonium sulphide T.S.

Tests for Identity.—Given above.

Impurities.—Sulphur, arsenic, antimony, and phosphorus (given above). Arsenic, cadmium, lead, and copper. Details in Part V.

Remarks.—As will be noted, the pharmacopœia permits the use of zinc in several forms, viz., as sticks, in the form of mossy zinc and sheet zinc, or in the form of a very fine powder. All these forms are used either for the manufacture of the official zinc salts or for the production of hydrogen. In the latter case zinc is largely used for the purpose of making arsenic tests, and it is essential that the metal so used should be free from arsenic and other impurities found largely in the ordinary commercial zinc. Hence it should be noticed that the pharmacopœia directs a careful test for the presence of arsenic in zinc.

Hydrogen can be produced from zinc in two ways:

Treatment of zinc with a diluted acid:



Treatment of zinc with a strong alkali:



These two reactions will be again mentioned in the chapter on Arsenic (p. 574), so here we need only touch upon that interesting compound, sodium zincate, Na_2ZnO_2 . Reference to the tests for zinc salts shows the fact that any soluble zinc salt,—say, zinc sulphate,—when treated with a solution of an alkali,—say, sodium hydroxide,—yields a white precipitate which will dissolve on addition of an excess of alkali.

The first stage of this reaction is as follows:

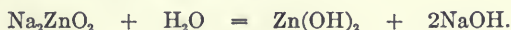


the white precipitate being zinc hydroxide.

When more sodium hydroxide is added to this, the precipitate is dissolved by reason of the formation of soluble sodium zincate by the following reaction:



Zincate of sodium, when largely diluted with water, decomposes, sodium hydroxide being produced, and zinc hydroxide being precipitated by the following equation:



Stress is here laid upon these combinations of zinc salts with alkalis because the same combinations occur when aluminum, manganese, chromium, and, to a lesser extent, iron, are treated with alkalis, this showing that such metals as zinc, aluminum, manganese, chromium, and even iron can act either as positive or negative elements, according as they are associated with elements more positive or negative than themselves.

Zinc is a valuable metal for use in the arts by reason of its power

of resistance against the atmospheric change. It is true that when a fresh layer of zinc is exposed to the air, it loses its lustre and becomes covered with a white layer. This layer consists of a mixture of zinc oxide and zinc carbonate, which acts as an admirable protective for the metal underneath, in a way similar to that already explained concerning the protective power of lead sulphate in the lead-chamber process. By reason of the superior stability of zinc, as compared with iron, it is quite largely used for the plating of iron utensils, such zinc-coated iron being called *galvanized iron*.

The tests for zinc and its compounds are: Ammonium sulphide produces a white precipitate of zinc sulphide when brought in contact with a solution of zinc salt. This explains the value of zinc oxide paint. The other chemical largely used as a white pigment is lead carbonate, and this so-called lead paint possesses the objection of blackening when coming in contact with hydrogen sulphide, that reagent thereby producing the black lead sulphide. Therefore, in places where hydrogen sulphide abounds, such as chemical laboratories or water-closets, the zinc paints are preferable.

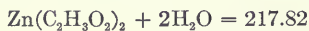
Any alkaline hydroxide produces a flocculent precipitate of zinc hydroxide with any soluble zinc salt, this precipitate redissolving in excess of alkali. Any soluble carbonate precipitates any zinc salt as a dense white zinc carbonate.

The following salts of zinc and their preparations are official:

Zinc acetate. At least 99.5 per cent. absolute $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 + 2\text{H}_2\text{O}$.
 Zinc bromide. At least 97 per cent. absolute ZnBr_2 .
 Precipitated zinc carbonate. Yielding at least 72 per cent. zinc oxide.
 Zinc chloride. At least 99.5 per cent. absolute ZnCl_2 .
 Solution of zinc chloride, containing 50 per cent. absolute ZnCl_2 .
 Zinc iodide. At least 98 per cent. absolute ZnI_2 .
 Zinc oxide. At least 99.5 per cent. absolute ZnO .
 Ointment of zinc oxide contains 20 per cent. zinc oxide.
 Zinc phenolsulphonate contains 99.5 per cent. absolute $\text{Zn}(\text{C}_6\text{H}_4\text{O}_3\text{S})_2 + 8\text{H}_2\text{O}$.
 Zinc stearate. $\text{Zn}(\text{C}_{18}\text{H}_{35}\text{O}_2)_2$ admixed with traces of zinc palmitate.
 Ointment of zinc stearate contains 50 per cent. zinc stearate.
 Zinc sulphate. At least 99.5 per cent. absolute $\text{ZnSO}_4 + 7\text{H}_2\text{O}$.
 Zinc valerate. At least 99 per cent. absolute $\text{Zn}(\text{C}_5\text{H}_9\text{O}_2)_2 + 2\text{H}_2\text{O}$.

Salts of zinc are usually made from the metal or from the oxide, by treatment with the proper acid. Zinc is a bivalent, even as are alkaline earths (p. 495).

ZINCI ACETAS. Zinc Acetate.



It should contain in the uneffloresced condition not less than 99.5 per cent. of pure Zinc Acetate $[(\text{CH}_3\text{COO})_2\text{Zn} + 2\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

Soft, white, six-sided, monoclinic plates, of a pearly lustre, having a faintly acetous odor, and in dilute solutions an astringent, metallic taste. Exposed to the air, the salt gradually effloresces and loses some of its acid.

Soluble in about 2.5 parts of water and in 36 parts of alcohol at 25°C . (77°F .); soluble in 1.5 parts of boiling water and in 0.6 part of boiling alcohol. On protracted boiling with water acetic acid is lost, and an insoluble basic salt formed.

When heated the salt is partially fused, losing water and acid. At a higher temperature it is decomposed, evolving acetone and other combustible vapors, and leaving a residue of zinc oxide.

Its aqueous solution reddens blue litmus-paper.

Tests for Identity.—See Zinc and Acetates in Part V.

Impurities.—Arsenic, cadmium, lead, copper, sulphate, and chloride. Details in Part V.

Remarks.—This salt is made by dissolving zinc oxide in acetic acid, as shown in the following equation:



Zinc acetate is a mild astringent, of considerable value in the manufacture of eye-washes and gonorrheal injections. For the latter purpose a favorite prescription is one combining lead acetate and zinc sulphate. When the solutions of these two salts are mixed, a double decomposition occurs in the formation of zinc acetate, which remains in solution, and lead sulphate, which precipitates out (p. 1080).

Zinc acetate is rarely given internally.

Dose.—125 milligrammes (2 grains).

ZINCI BROMIDUM. Zinc Bromide.



It should contain, when anhydrous, at least 97 per cent. of pure Zinc Bromide and should be kept in small, glass-stoppered bottles.

A white, granular powder; odorless, having, even in dilute solutions, a sharp, saline, and metallic taste. Very deliquescent.

Readily soluble in water and alcohol.

When heated to 394° C. (741.2° F.) the salt fuses, and, with a careful increase of heat, may be sublimed in the form of needle-shaped prisms.

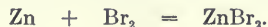
Its aqueous solution gives a slightly acid reaction with blue litmus-paper.

Tests for Identity.—See Zinc and Bromides in Part V.

Impurities.—Iodide, arsenic, cadmium, lead, copper, sulphate, excess of chloride. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by either one of two ways: A solution of zinc sulphate is treated with a solution of potassium bromide, the potassium sulphate separated from the solution of zinc bromide by the addition of alcohol (see Ammonium Iodide, p. 493). A better method, however, is by treating bromine placed under a considerable layer of water with granulated zinc, the reaction being as follows:



In both cases the salt is obtained from the solution by granulation.

Medical Properties.—Zinc bromide is used as a hypnotic in epilepsy.

Dose.—125 milligrammes (2 grains).

ZINCI CARBONAS PRÆCIPITATUS. Precipitated Zinc Carbonate.

Hydrated Zinc Carbonate, which, upon ignition, should yield not less than 72 per cent. of zinc oxide [$\text{ZnO} = 80.73$].

An impalpable, white powder, of somewhat variable chemical composition, without odor or taste. Permanent in the air.

Insoluble in water or alcohol; completely soluble in diluted acids with copious effervescence; also soluble in ammonia water and in ammonium carbonate T.S.

When strongly heated the salt loses water and carbon dioxide, and leaves a residue, which is yellow while hot, but becomes white on cooling.

When a small portion of the salt is moistened with a drop of cobaltous nitrate T.S., and heated before the blow-pipe, it will assume a vivid green color.

Tests for Identity.—See Zinc and Carbonates in Part V.

Impurities.—Arsenic, cadmium, lead, copper, and excess of alkali. Details in Part V.

Assay.—Details in Part V.

Remarks.—It will be noticed in the official definition given above that no chemical formula is assigned this salt, and this is because it is of variable composition.

It, like the official magnesium carbonate and lead carbonate, is a mixture of the true carbonate with the hydrate. Like magnesium carbonate, the proportion of the carbonate to the hydrate depends on conditions of precipitation—on concentration of solutions, temperature, and proportion of the two chemicals used in manufacture.

The official carbonate is made by mixing hot solutions of zinc sulphate with sodium carbonate. If cold diluted solutions of the two salts are mixed, the normal carbonate is produced, but during the reaction considerable carbonic acid is evolved, and the carbonate thus produced dissolves in this carbonated water. Hence the process is wasteful, and in factories recourse is always had to the process of hot precipitation. The normal carbonate, moreover, is unstable, producing, on drying, the official carbonate.

Zinc carbonate is used chiefly externally as a dusting-powder, being mildly astringent.

ZINCI CHLORIDUM. Zinc Chloride.



It should contain, when anhydrous, not less than 99.5 per cent. of pure Zinc Chloride and should be kept in small, glass-stoppered bottles.

A white, granular powder, or porcelain-like masses, irregular, or molded into pencils; odorless, *of such intensely caustic properties as to make tasting dangerous* unless the salt be dissolved in much water; the dilute solution has an astringent, metallic taste. Very deliquescent.

Soluble in about 0.4 part of water at 25° C. (77° F.), forming a clear solution, which, on protracted boiling, deposits a basic salt; very soluble in alcohol.

When heated to 115° C. (239° F.) Zinc Chloride fuses to a clear liquid. At a higher temperature it is partly volatilized in dense, white fumes, and partly decomposed, leaving a residue of zinc oxide.

Its aqueous solution reddens blue litmus-paper.

The aqueous solution (1 in 20) should be clear, or at most only very slightly opalescent; and if it be mixed with an equal volume of alcohol, a single drop of hydrochloric acid should suffice to render 10 Cc. of the mixture perfectly clear (limit of *oxychloride*).

Tests for Identity.—See Zinc and Chlorides in Part V.

Impurities.—Excess of oxychloride (given above). Arsenic, cadmium, lead, copper, sulphates. Details in Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by treating metallic zinc with hydrochloric acid, as shown in the following equation:



The solution of chloride thus produced is evaporated to dryness and placed in warm dry bottles.

Zinc chloride is used chiefly externally as an antiseptic, disinfectant, and caustic. For the latter purpose it enjoyed considerable repute in cases of cancer, a preparation largely used in France—

paté de Canquoin—being a pasty mass of flour with which is incorporated zinc chloride.

LIQUOR ZINCI CHLORIDI. Solution of Zinc Chloride.

Complete details of the manufacture of this solution, called *Burnett's disinfecting fluid*, including the official recipe, will be given in Part VII., so here we need only mention that it is made by treating zinc with hydrochloric acid, evaporating the solution to dryness, adding to the residue a small quantity of nitric acid, fusing the mass, and then redissolving it in sufficient cold water to make the official liquid contain 50 per cent. of zinc chloride. The addition of nitric acid in fusing is done in order to insure the removal of traces of iron which invariably contaminate the commercial metallic zinc, and in this preparation the latter is usually employed, instead of the official chemically pure metal. Under the treatment just given the zinc chloride undergoes no change, but the iron chloride will be converted into Fe_2O_3 , which is insoluble in water (p. 1124).

ZINCI IODIDUM. Zinc Iodide.

$$\text{ZnI}_2 = 316.70$$

It should contain, when anhydrous, not less than 98 per cent. of pure Zinc Iodide and should be kept in small, glass-stoppered bottles, protected from light.

A white, granular powder; odorless and having a sharp, saline, and metallic taste. Very deliquescent, and, upon exposure to air and light, becoming brown from liberated iodine.

Readily soluble in water, alcohol, or ether.

When heated to about 446°C . (834.8°F .) the salt fuses to a colorless liquid, and at a higher temperature sublimes, forming quadratic needles, while a small part is decomposed and leaves a residue of zinc oxide.

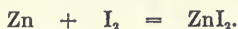
Its aqueous solution reddens blue litmus-paper.

Tests for Identity.—See Zinc and Iodides in Part V.

Impurities.—Arsenic, cadmium, lead, copper, sulphate, and excess of chloride. Details in Part V.

Assay.—Details in Part V.

Remarks.—This is made by combining metallic zinc with iodine, as shown in the following equation:



This can be done by combining the two elements dry and subliming the resulting zinc iodide, or the elements can be digested under water and zinc iodide obtained in crystals by evaporation of the solutions.

This salt is used as an alterative.

Dose.—65 milligrammes (1 grain).

ZINCI OXIDUM. Zinc Oxide.

$$\text{ZnO} = 80.78$$

It should contain not less than 99.5 per cent. of pure Zinc Oxide.

A very fine, amorphous, white or yellowish-white powder, free from gritty particles, without odor or taste; it gradually absorbs carbon dioxide from the air.

Insoluble in water or alcohol. Completely soluble, without effervescence, in diluted acids; also in ammonia water and in ammonium carbonate T.S.

When heated, it assumes a yellow color, which disappears on cooling.

If a small portion of Zinc Oxide be moistened with a drop of cobaltous nitrate T.S., and heated before the blow-pipe, it will assume a vivid green color.

Tests for Identity.—Given above.

Impurities.—Arsenic, cadmium, lead, copper, excess of alkali, excess of sulphate, and chloride. Details in Part V.

Assay.—Details in Part V.

Remarks.—This is prepared by the calcination of pure zinc carbonate. It is important for the pharmacist to understand the distinction between the commercial zinc oxide, which is so largely used, and the form recognized by the pharmacopœia. The commercial form is not made by the calcination of zinc carbonate, but by the roasting of crude metallic zinc in a current of air, whereby the resulting zinc oxide sublimes. This form of zinc oxide is used very largely as a paint, and should never be used by the careful pharmacist in making the official zinc salve.

Commercial zinc oxide is of dead white tint, and is more or less gritty, while the official compound has a slightly cream tint. Were it universally employed by the pharmacist for making the official zinc ointment, there would be less complaint about the grittiness of that preparation.

Two synonyms of zinc oxide are worthy of passing notice as relics of the days of alchemy. The chemical is sometimes called *lana philosophica* (philosopher's wool), because the sublimed zinc oxide collects on the condenser in wooly tufts. The other synonym, *nihilum album*, is an interesting illustration of corruption in words in the course of time. The Latin words *nix* and *album* (white snow) were coined by some poetic alchemist because of the similarity of sublimed zinc oxide to snow-flakes. The term, in being used by another author, was misunderstood and written as "nichts album" (white nothing), which term was found in several books of the seventeenth century. Later some careful student decided the term "nichts album," combining as it did German and Latin, should be placed in correct Latin, and thus the form "*nihilum album*" (white nothing) originated.

Zinc oxide is used almost entirely externally, and so used it is a mild, healing astringent. It is chiefly applied in the form of the official ointment. (See p. 349). If used internally, it is an astringent.

Dose.—250 milligrammes (4 grains).

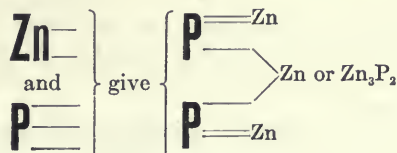
OLEATUM ZINCI (U. S. P. 1890). Oleate of Zinc.

Oleate of zinc (U. S. P. 1890) was made by sifting zinc through a fine sieve into oleic acid contained in an evaporating dish, and warming until the oxide was dissolved. As already explained under the head of Oleates, this chemical can be considered as a pharmaceutical—as a solution of zinc oleate in a large excess of oleic acid. It is referred to here in order to emphasize the distinction between it and the true *zinc oleate*. While the official oleate of zinc is in the form of a thin, greasy liquid, the definite chemical zinc oleate is a smooth white powder, and is made by mixing solutions of Castile soap and zinc sulphate. A recipe of this form of zinc oleate can be found in the National Formulary. It is used in a manner similar to zinc oxide, but by some specialists is preferred to the latter for dusting purposes.

ZINCI PHOSPHIDUM (U. S. P. 1890). Zinc Phosphide.

Zinc phosphide (U. S. P. 1890) was made by passing the vapor of phosphorus over powdered zinc in an atmosphere of hydrogen. The performance of this operation, it is needless to say, requires the utmost caution. The zinc is placed in a tube

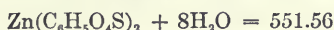
of Bohemian glass, the air having been carefully excluded by passing hydrogen through the tube. The phosphorus is then distilled from the retort, from which the air has also been removed by means of hydrogen, and under such circumstances the two elements combine without danger. Zinc phosphide affords an interesting problem of linking by valence:



It is nearly five times heavier than water.

Zinc phosphide is a nervous stimulant and aphrodisiac, and is administered in doses of from 3 to 8 milligrammes.

ZINCI PHENOLSULPHONAS. Zinc Phenolsulphonate.



It should contain, in uneffloresced crystals, not less than 99.5 per cent. of pure Zinc Paraphenolsulphonate $[(\text{C}_6\text{H}_4(\text{OH})\text{SO}_3)_2\text{Zn} 1:4 + 8\text{H}_2\text{O}]$ and should be kept in small, well-stoppered bottles.

Colorless, transparent, rhombic prisms or tabular crystals; odorless and having an astringent, metallic taste. Exposed to the air the salt effloresces, and upon exposure to light and air may become slightly pink.

Soluble in 1.7 parts of water or alcohol at 25° C. (77° F.); in 0.3 part of boiling water and 0.56 part of boiling alcohol.

When heated to 100° C. (212° F.) the salt loses 6 molecules of water of crystallization, and it loses the remainder at 125° C. (257° F.). At a higher temperature it chars, emitting inflammable vapors having the odor of phenol, and finally leaves a residue amounting to about 14.6 per cent. of the original weight.

Its aqueous solution reddens blue litmus-paper.

Tests for Identity.—See Zinc and Phenolsulphonates in Part V.

Impurities.—Arsenic, cadmium, lead, copper, sulphate, and chloride. Details in Part V.

Remarks.—This new official, also called *zinc sulphocarbolate*, is made by treating barium sulphocarbolate with zinc sulphate, when barium sulphate precipitates, leaving zinc sulphocarbolate in solution. By evaporation of this solution the salt is obtained in crystals. The manufacture of barium sulphocarbolate has already been described on p. 469.

Zinc phenolsulphonate is a useful astringent antiseptic.

Dose.—125 milligrammes (2 grains).

ZINCI STEARAS. Zinc Stearate.

A very fine, white powder, tasteless, and having a very faint odor, resembling that of fat. Zinc stearate contains a small but varying proportion of zinc palmitate. Insoluble in water, alcohol, or ether.

When heated, the salt fuses. At a higher temperature it is decomposed, giving off inflammable vapors and the odor of burning fat, and finally leaves about 15.5 per cent. of residue, which consists chiefly of zinc oxide.

The salt should have a neutral reaction to moistened litmus-paper.

If 0.5 Gm. of Zinc Stearate be heated with a mixture of 9.5 Cc. of distilled water and 0.5 Cc. of hydrochloric acid, stearic acid will be liberated and float as an oily layer on the surface of the liquid.

If, after filtering this liquid through a small wetted filter, all of the zinc be precipitated by ammonium sulphide T.S., the filtrate should leave no fixed residue on evaporation (absence of *alkalis*, *alkali earths*, etc.).

If 1 Gm. of Zinc Stearate be boiled with 50 Cc. of distilled water containing 2 Cc. of nitric acid, and filtered through a wetted filter, after thoroughly washing the precipitate with boiling water, the filtrate and washings, when evaporated to dryness and ignited, should leave a residue weighing not less than 0.14 Gm. and not more than 0.16 Gm.

Tests for Identity.—Given above.

Impurities.—Alkalis and alkaline earths (given above). Excess of chlorides. Details in Part V.

Assay.—Given above.

Remarks.—This new official is a combination of zinc stearate with traces of zinc palmitate. It is made by treating a solution of sodium stearate with a solution of zinc acetate or sulphate, collecting the precipitate, and washing carefully. The sodium stearate is made by treating stearic acid with a hot solution of sodium carbonate.

The resulting white powder is used for dusting purposes, and in the form of ointment. A 50 per cent. ointment is now official. (See p. 350.)

ZINCI SULPHAS. Zinc Sulphate.



It should contain, in uneffloresced crystals, not less than 99.5 per cent. of pure Zinc Sulphate [$\text{SO}_4, \text{O}_2\text{Zn} + 7\text{H}_2\text{O}$] and should be kept in well-stoppered bottles.

Colorless, transparent, rhombic crystals, or a granular crystalline powder, without odor, and having an astringent, metallic taste. Efflorescent in dry air.

Completely soluble in 0.53 part of water at 25° C. (77° F.) and in 0.2 part of boiling water; soluble in about 3 parts of glycerin; insoluble in alcohol.

When rapidly heated the salt melts. At a higher temperature it is partly decomposed, losing both water and sulphuric acid. When very gradually heated to 50° C. (122° F.) it loses 5 molecules of its water of crystallization (31.2 per cent.) without melting. At 100° C. (212° F.) a sixth molecule is lost, while the last is removed, with decomposition of the salt, at a temperature of about 240° C. (464° F.).

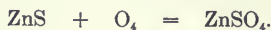
Its aqueous solution shows an acid reaction with blue litmus-paper.

If 1 Gm. of Zinc Sulphate, in small fragments, be agitated for some time with 10 Cc. of alcohol, the filtrate should not redden moistened blue litmus-paper (absence of free acid).

Tests for Identity.—See Zinc and Sulphates in Part V.

Impurities.—Arsenic, cadmium, lead, copper, excess of chloride. Details in Part V. Free acid, given above.

Remarks.—Zinc sulphate (or *white vitriol*) is made on a large scale by roasting the ore zinc blend (zinc sulphide). The oxygen of the air combines with the heated zinc sulphide, forming zinc sulphate, as shown in the following equation:



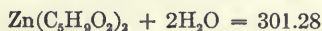
The sulphate is separated from the extraneous mass of silica and other metals by lixiviation and is then crystallized.

It is also made by treating zinc oxide or metallic zinc with sulphuric acid. Commercial zinc contains iron and other metals, and these are removed from the acid solution by digesting with an excess of zinc, saturating the solution with chlorine gas, and then adding precipitated zinc carbonate. The filtered solution is then evaporated to drive off chlorine and to concentrate to the point at which the sulphate will begin to crystallize.

Zinc sulphate is a very valuable astringent, for which purpose it is given internally in doses of from 0.06 to 0.12 Gm. In doses five times as great it is an emetic, and is used considerably in evacuating the stomach in cases of poison. For ordinary purposes of emesis it is too irritating, and ipecac is preferable. Externally, zinc sulphate solution is used as an astringent wash, particularly in cases of gonorrhea.

Dose.—As emetic, 1 Gm. (15 grains).

ZINCI VALERAS. Zinc Valerate.



[ZINCI VALERIANAS, PHARM. 1890]

It should contain not less than 99 per cent. of pure Zinc Valerate $[(\text{C}_5\text{H}_9\text{COO})_2\text{Zn} + 2\text{H}_2\text{O}]$ and should be kept in small, well-stoppered bottles.

White, pearly scales, having the odor of valeric acid, and a sweetish, astringent, and metallic taste. On exposure to the air, it slowly loses valeric acid.

Soluble in about 50 parts of water and in about 35 parts of alcohol at 25° C. (77° F.); somewhat more soluble in absolute alcohol. Boiling renders the aqueous solution turbid from loss of acid and formation of a basic salt.

When heated, the salt melts. At a higher temperature it is decomposed, giving off inflammable vapors, and finally leaving a residue of zinc oxide.

Its aqueous solution reddens blue litmus-paper.

If 0.5 Gm. of Zinc Valerate be dissolved in a mixture of 0.5 Cc. of hydrochloric acid and 9 Cc. of water, the valeric (isovaleric) acid will be liberated and float as an oily layer on the surface of the liquid.

Tests for Identity. Given above.

Impurities.—Arsenic, cadmium, lead, copper, sulphate, chloride, acetate, and butyrate. Details in Part V.

Remarks.—Zinc valerianate is one of the two official salts of valerianic acid (p. 688). It may be made by a double decomposition between sodium valerianate and zinc sulphate.

Zinc valerianate is a valuable nerve and antispasmodic.

Dose.—125 milligrammes (2 grains).

MANGANESE

Symbol, Mn. Atomic weight, 54.6

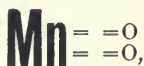
The chief ores of manganese are *pyrolusite* (MnO_2), *braunite* (Mn_2O_3), *hausmannite* (Mn_3O_4), and *manganese spar* (MnCO_3).

Manganese affords several official salts. A word as to the origin of its name, which is similar to another element, magnesium, and to the term magnet. In the days of Pliny, at the beginning of the Christian era, three products bearing the name *magnesia* were known, all coming either from the province of Magnesia, on the borders of Macedonia, or from the country around the city of Magnesia in Asia Minor. Pliny describes all three. One was white (our present magnesium carbonate), one was black and attracted iron (magnetic iron ore), while a third was brown and was used in glass making (manganese dioxide). That form attracting iron was rechristened magnet before the days of Avicenna (A. D. 980–1037), for in his medical writings we find a chapter on magnets, entitled “de Magnete.” The distinction in the name manganese and magnesium was not made until the beginning of last century, after both elements had been isolated.

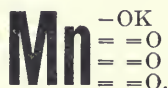
Manganese is obtained by the reduction of the oxide with charcoal at red heat.

It is a gray-white, hard metal, having the specific gravity of 7.2. It is, like zinc, comparatively stable in air, although it does oxidize in moist air, and more quickly when immersed in water.

Manganese is an element whose valence shows interesting variation. In manganese sulphate, MnSO_4 , its valence seems to be II; in manganese dioxide, MnO_2 , the valence of the metal is IV—

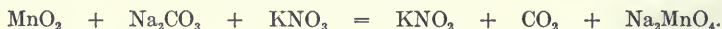


while in potassium permanganate, KMnO_4 , manganese shows the valence VII; the graphic formula of the compound being—



Manganese, like zinc, seems to occupy a position between the non-metallic and the metallic elements. It has been shown that when zinc is combined with strong alkalis, zincates are formed, the zinc hydrate in that case acting as an acid. Still more striking is the case with manganese oxides, particularly those showing the valence VI and VII, permanganic acid, HMnO_4 , being as distinctly acid as is perchloric acid (HClO_4). Yet the manganese with lower valence has basic properties, and when the metal is treated with acid, it yields a manganese salt in which the element shows the valence II.

Tests.—Manganese is interesting to the chemist as affording a large variety of very beautiful tests. When a soluble salt of manganese is treated with ammonium sulphide solution, a flesh-colored precipitate of manganese sulphide is produced. This precipitate is notable as the only flesh-colored sulphide. If a trace of manganese salt is added to a borax bead (see p. 985), a beautiful amethyst color is transmitted to the same. If any manganese salt is fused with a mixture of potassium nitrate and sodium carbonate on a piece of platinum foil, green sodium or potassium manganate is formed, as shown in the following typical equation:



If this green manganate be boiled with water, a pink solution of the permanganate is formed by the following reaction:



Crum's test also exhibits the same purple color, due to permanganic acid. It is as follows: if a manganese salt is treated with plumbic oxide and nitric acid, permanganic acid is formed, as shown in the following equation:



The same reaction occurs if red lead or if litharge is used instead of plumbic oxide.

The following compounds of manganese are official:

Precipitated manganese dioxide. At least 80 per cent. absolute MnO_2 .
 Manganese hypophosphite. At least 97 per cent. absolute $\text{Mn}(\text{PH}_2\text{O}_2)_2 + \text{H}_2\text{O}$.
 Manganese sulphate. At least 99.5 per cent. absolute $\text{MnSO}_4 + 4\text{H}_2\text{O}$.

To which might be added

Potassium permanganate, considered among the potassium salts.

MANGANI DIOXIDUM PRÆCIPITATUM. Precipitated Manganese Dioxide.

[To replace MANGANI DIOXIDUM, PHARM. 1890]

Chiefly Manganese Dioxide [$\text{MnO}_2 = 86.36$] with small amounts of other oxides of manganese, corresponding to not less than 80 per cent. of Manganese Dioxide.

Recipe.—Manganese Sulphate, fifty grammes..... 50 Gm.
 Ammonia Water, two hundred and fifty cubic centimeters..... 250 Cc.
 Solution of Hydrogen Dioxide, two hundred and fifty cubic centimeters..... 250 Cc.
 Distilled Water, a sufficient quantity.

Dissolve the Manganese Sulphate in *one thousand cubic centimeters* of Distilled Water. Dilute the Ammonia Water with an equal volume of Distilled Water, and mix it with the Solution of Hydrogen Dioxide, which has also been diluted with an equal volume of Distilled Water. Pour the mixed solutions slowly, with constant stirring, into the solution of Manganese Sulphate. Allow the mixture to stand for one hour, stirring frequently. Then decant the supernatant clear liquid from the precipitate, and wash the latter repeatedly by affusion and decantation with hot Distilled Water, using *one thousand cubic centimeters* each time. Collect the precipitate on a plain filter, and continue the washing with hot Distilled Water until the washings no longer have an alkaline reaction upon red litmus-paper and produce no turbidity when mixed with barium chloride test solution. Allow the precipitate to drain, then dry it at 150°C . (302°F .).

A heavy, very fine, black powder, without odor or taste; permanent in the air.

Insoluble in water or alcohol. It is not affected by concentrated sulphuric acid, but when heated with this acid it is converted into manganous sulphate, with the evolution of oxygen.

When heated with hydrochloric acid, it is converted into manganous chloride, with the evolution of chlorine. At a red heat Precipitated Manganese Dioxide gives off oxygen, and is converted into reddish-brown manganoso-manganic oxide (Mn_2O_3).

On intimately mixing 1 part of the Dioxide with 1 part of potassium hydroxide and 1 part of potassium chlorate, introducing the mass into a crucible, moistening with water, drying and igniting, a dark fused mass is obtained, which yields, with water, a green solution, changing to purplish-red on being boiled or on the addition of diluted sulphuric acid.

If to 1 Gm. of the Dioxide and 2 Gm. of oxalic acid, 20 Cc. of water be added, followed by 3 Cc. of sulphuric acid, and the mixture digested for several hours on a water-bath, complete solution should be affected (absence of *antimony sulphide* and *insoluble substances*).

Tests for Identity.—Given above.

Impurities.—Antimony sulphide and insoluble substances. Given above.

Assay.—Details in Part V.

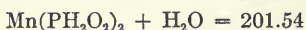
Remarks.—This chemical, the recipe for the manufacture of which is just given, replaces the crude ground ore, pyrolusite, which was recognized in the former pharmacopœia. The new official is made

by precipitating manganous sulphate with ammonia water in the presence of hydrogen dioxide solution. Were manganous sulphate precipitated with ammonia water alone, manganous oxide (MnO) would result; hence the use of the oxidizing agent, hydrogen dioxide.

Precipitated manganese dioxide is used for anemia, particularly that form which comes from female complaints. It is administered preferably by filling capsules with the prescribed amount of the binoxide in fine powder.

Dose.—250 milligrammes (4 grains).

MANGANI HYPOPHOSPHIS. Manganese Hypophosphite.



It should contain not less than 97 per cent. of pure Manganous Hypophosphite [$(\text{PH}_2\text{O}_2)_2\text{Mn} + \text{H}_2\text{O}$] and be kept in well-stoppered vials.

A pink crystalline powder, odorless, and nearly tasteless; permanent in the air.

Soluble in 6.6 parts of water at 25° C. (77° F.) and in 6 parts of boiling water; almost insoluble in alcohol.

The aqueous solution of the salt (1 in 20) is neutral to litmus-paper, and yields, with ammonium sulphide T.S., a salmon-colored precipitate of manganese sulphide, soluble in acetic acid.

Tests for Identity.—See Manganese and Hypophosphites in Part V.

Impurities.—Carbonate, phosphate, calcium, and arsenic. Details in Part V.

Remarks.—This new official is made by mixing a solution of manganese oxalate with a solution of calcium hypophosphite and evaporating the filtrate on a water-bath to a granular powder.

It is a constituent of the new official preparation, compound syrup of hypophosphites, and is used as a tonic.

Dose.—20 milligrammes (3 grains).

MANGANI SULPHAS. Manganese Sulphate.



It should contain not less than 99.5 per cent. of pure Manganous Sulphate [$\text{SO}_4\text{O}_2\text{Mn} + 4\text{H}_2\text{O}$] and should be kept in well-stoppered bottles.

Colorless, or pale rose-colored, translucent, tetragonal prisms (crystallized at a temperature between 20° and 30° C. (68° and 86° F.), and containing 4 molecules, or 32.29 per cent. of water of crystallization); odorless and having a slightly bitter and astringent taste. Slightly efflorescent in the air.

Soluble in about 0.7 part of water at 25° C. (77° F.) and in 0.53 part of boiling water; insoluble in alcohol.

The aqueous solution (1 in 20) is neutral or very slightly acid to litmus-paper.

If the manganese be completely precipitated from an aqueous solution of 3 Gm. of the salt by the addition of ammonium carbonate T.S., the filtrate, on evaporation and gentle ignition, should leave no weighable residue (absence of *salts of the alkalis*, and of *magnesium*).

If 1 Gm. of the salt be gently ignited, in a porcelain crucible, it should lose not more than 0.323 Gm. of its weight (distinction from *Manganese Sulphate containing a larger amount of water of crystallization*).

Tests for Identity.—See Manganese and Sulphates in Part V.

Impurities.—Iron, heavy metals, and zinc. Details in Part V. Salts of alkalis and of magnesium, excess of water of crystallization. Details given above.

Remarks.—This salt is produced by treating the binoxide of

manganese with carbon at red heat, whereby it is reduced to manganous oxide, MnO , as shown in the following equation:



The manganous oxide is made into paste with sulphuric acid and heated to redness, when manganese sulphate is formed, as shown in the following equation:



and goes into solution and is purified by crystallization.

The sulphate and the hypophosphite of manganese are easy to recognize because they are the only official salts occurring in rose-colored crystals.

Manganese sulphate is used as a tonic, similar to the iron preparation, and is also used for gout.

Dose.—250 milligrammes (4 grains).

POTASSII PERMANGANAS. Potassium Permanganate.

This salt has been considered under the potassium compounds (see p. 451), so here we need only again call attention to the synonym, *mineral chameleon*, it being so called because of the changes in tint which the permanganate exhibits under treatment with various reagents.

COBALT AND NICKEL

As neither of these metals, nor any of their compounds, is recognized by the pharmacopœia, they can be briefly considered together.

Cobalt, so called from the German *Kobold*, because the miners working in cobalt ores found the same difficult to extract, and attributed this difficulty to the influence of the kobolds, mischievous imps supposed to inhabit mines, occurs in the form of a silvery metal, but little used in the arts. Of its compounds, the silicate is of a striking blue color, and is used in painting and decorating under the name of *smalt*; the chloride, CoCl_2 , occurs in the form of pink crystals, which comparatively easily effloresce and in so doing will change to a blue tint. For this reason the solution of cobalt chloride has been suggested for *invisible ink* or *sympathetic ink*, the writing appearing practically colorless until heated, when the effloresced salt develops itself in blue tracery. It is also used in the manufacture of cheap paper barometers, consisting of bibulous paper dipped into a solution of cobalt chloride and dried. In moist weather this paper appears to be pink, whereas on drying it becomes blue through the efflorescence of the salt.

Metallic nickel, on the other hand, is very largely used. It is of value for cooking utensils, although for this purpose it is somewhat costly, while the use of nickel coins is so extended as to require no additional comment. The coating of utensils with the metal by electric deposition (*nickel-plating*) is also well known, nickel being used in such cases because it does not easily tarnish.

A valuable alloy of nickel is *German silver*. This consists of 50 per cent. of copper and 25 per cent. each of zinc and of nickel. This alloy possesses a silvery tint, and was formerly largely used as the metallic trimming of glass show-cases.

German silver is the first alloy to which we have referred, and it might be here stated that by an *alloy* we mean a solution of several metals with each other while in a fused state. Such alloys frequently possess valuable properties not shown by their constituents, and many alloys of great value will be discussed on later pages.

CHAPTER XXX

ALUMINUM, CERIUM, AND CHROMIUM

In the last chapter (p. 517) it was stated that the third and fourth groups into which the metals are divided in analytic chemistry include all those elements precipitated from alkaline solution by ammonium sulphide.

It was also learned that the third group were those elements whose hydrates redissolved in excess of ammonium hydrate; while the hydrates of the fourth group, aluminum, iron, cerium, and chromium, did not dissolve in ammonia water. These four metals form, even as did the metals discussed in the last chapter, a group that should be included in one chapter. Since, however, the compounds of iron used in medicine are so numerous and valuable, it is deemed best to consider these in a special chapter, leaving aluminum, cerium, and chromium to be discussed in this chapter.

ALUMINUM

Symbol, Al. Atomic weight, approximately 27.

Aluminum is one of the most widely distributed of metals, being found in the form of silicate in *clay*, which is one of the most abundant constituents of the earth's crust. A rare and valuable form of the silicate is sodio-aluminum sulphosilicate, the precious mineral, *lapis lazuli*, while among the oxides of aluminum are found several of the most valuable precious stones, such as *ruby* (which, next to diamond, is the hardest of minerals) and *sapphire*. The color of the ruby is due to a trace of chromium, while the sapphire is tinted with cobalt. *Emerald* is a silicate of aluminum and beryllium, with a trace of chromium sesquioxide (Cr_2O_3). *Emery* is a native granular form of aluminum oxide.

While the salts of aluminum, especially alum, have been known from ancient times, the metal was first isolated by Friedrich Wöhler, in 1827, by heating aluminum chloride with metallic potassium in a well-closed crucible.

Wöhler soon found he could obtain the same results by the use of the cheaper metal, sodium. Ever since its isolation by Wöhler, efforts have been made to devise a cheap process of manufacture, since its lightness, toughness, and, above all, its disinclination to oxidize—to rust, as iron does—gave promise of its great value in the arts.

Metallic potassium and sodium are costly metals; hence aluminum so separated could never compete, in price, with iron. Experiments in this direction, however, especially those performed under the patronage of Louis Napoleon, while emperor, led to the methods of extraction of the metals sodium and potassium, devised by St. Clair Deville, and a marked reduction in the price of these metals.

Aluminum, one of the most abundant of elements, found in the very rocks and clay beneath our feet, is also one of the most difficult to isolate, and it was not until 1886—over half a century after its first isolation by Wöhler—that a cheap method of obtaining the metal

was devised, and that by two Americans—Messrs. Cowles, of Cleveland. This method is by electrolysis.

Already, on p. 366, we learned that molten salt, when acted upon by an electric current, is so decomposed that the positive element, sodium, goes to the negative pole, while the negative, chlorine, goes to the positive pole, of the battery. This process Messrs. Cowles applied to the separation of aluminum from its oxide, emery, and that on a large scale. The process operates on the same principle as an electric arc-light, which is produced by passing a powerful current of electricity through carbon rods representing the positive and negative poles of the battery or dynamo. In the Cowles' process a powerful current is run through two bundles of carbon rods several feet long, plunged in a mixture of emery and coke. The great heat produces a fusion of the oxide, which is decomposed by the current into the metal. The metal thus obtained is quite impure, and this process is now used chiefly for making alloys of aluminum, such as those with copper. For obtaining the pure metal, the Heroult process and the Hall process are employed. These processes were introduced about the same time, and are almost identical; in both cases a mixture of cryolite and aluminum oxide are fused, extra aluminum oxide being added from time to time to make up for that removed as the metal, thus making the process practically continuous.

Regardless of the present value of the Cowles' process, the fact remains that their discovery revolutionized the aluminum industry by bringing the price of the metal to a figure permitting its general use. To Americans the Cowles' discovery should be a matter of peculiar satisfaction, inasmuch as through it the center of aluminum production was shifted from Germany and Switzerland to America.

The following table, as to reduction in price of aluminum, can be best understood if one remembers that a kilo is 2½ pounds and a franc is 20 cents; that the decrease in market price between 1855 and 1856 was due to the introduction of the St. Clair Deville process for making cheap sodium; while the fall in price between 1886 and 1890 was due to the introduction of the Cowles' process.

The other reductions are in natural sequence to the gradual improvements to these two important processes.

MARKET PRICE PER KILOGRAMME OF ALUMINUM

1855—1250 francs.	1886—125 francs.	1891—6 francs.
1856— 375 “	1890— 19 “	1895—4 “
1857— 300 “		1898—2.70 “

ALUMINUM PRODUCTION (AMOUNTS IN KILOS)

YEAR.	UNITED STATES, ENGLAND, AND FRANCE.		GERMANY AND SWITZERLAND.	
1885.....	3000		10,292	
	UNITED STATES.	ENGLAND.	FRANCE.	SWITZERLAND.
1890.....	27,000	70,000	37,000	40,538
1891.....	1,800,000	300,000	500,000	800,000
1898.....	2,358,000	300,000	500,000	800,000

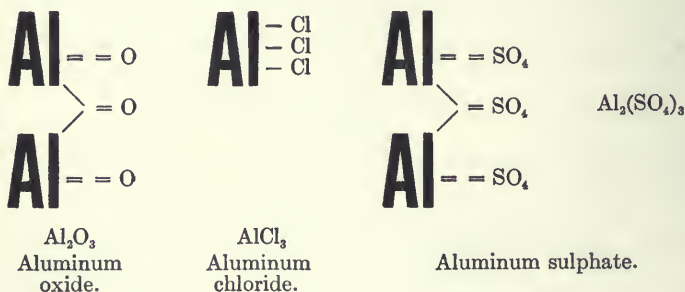
It will be noted that, while the world's output of aluminum in 1885 was 13,292 kilos, by 1898 its increase was so enormous that the production was 3,958,704 kilos.

Aluminum is a bright, silvery metal, having the specific gravity of 2.7. It is not easily oxidized, and on account of its comparative toughness and great lightness it was expected to play an important

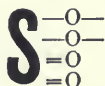
rôle in our metallic industries. Enthusiasts even predicted the eventual substitution of aluminum for iron in much structural work. Experience, however, has not substantiated the claims of the friends of aluminum, it being found that the tensile strength of aluminum was quite low. When worn, it becomes decidedly brittle. It was also hoped that aluminum would be of considerable service in chemical utensils where platinum must now be used, such as in distillations. This, however, was not borne out by facts. Therefore the chief use of aluminum at the present time is for trinkets and ornaments, and, to a certain extent, for cooking utensils, its lightness rendering them far preferable to those made of iron. It is also a constituent of many valuable alloys, such as aluminum bronze, while almost all fine analytic balances have beams of aluminum.

Tests.—The tests for aluminum are, first of all, the white precipitate obtained by treating a soluble aluminum salt with ammonium sulphide, the precipitate in this case, however, not being aluminum sulphide, but aluminum hydroxide. A similar precipitate of the flocculent hydroxide is obtained by treating an aluminum salt with any soluble hydroxide. The behavior of this hydroxide with the reagent, however, differs if sodium or potassium hydroxide is used, on the one hand, or ammonium hydrate, on the other, the precipitate redissolving with an excess of potassium or sodium hydroxide, with the formation of the aluminates of sodium and potassium, $\text{Al}(\text{ONa})_3$ or $\text{Al}(\text{OK})_3$. The composition of these aluminates is discussed in the chapter on Sodium (p. 463). On the other hand, aluminum hydroxide does not redissolve in an excess of ammonia water.

Aluminum is the first metal we have studied having the valence III; hence it is well to consider its combinations. One atom replaces three atoms of hydrogen in an acid, as shown by the graphic formulas given below:



It will be noticed in the graphic formula of aluminum sulphate given above that the SO_4 group is represented as attaching itself to the aluminum atom with two bonds. That is, if two atoms of hydrogen are removed from H_2SO_4 , we have a radicle, SO_4 , with two free bonds, viz.:



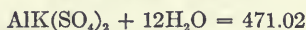
As these two free bonds act exactly as any other free bonds, the SO_4 group in formula-writing can be considered exactly as we consider an oxygen atom with its two bonds.

It should be stated in passing that many modern chemists think that all aluminum compounds contain two atoms of that element exhibiting the valence VI, and that the formula of aluminum chloride is not AlCl_3 , given above, but Al_2Cl_6 . This idea is based on the molecular weight estimations, for while AlCl_3 would mean molecular weight 133.5, the estimations give figures much nearer 267. The double formula, Al_2Cl_6 , is explained as is the formula Fe_2Cl_6 , which will be considered in the next chapter (p. 548).

The following compounds of aluminum are official:

Alum. At least 99 per cent. absolute $\text{AlK}(\text{SO}_4)_2 + 12\text{H}_2\text{O}$.
 Exsiccated alum. At least 99 per cent. absolute $\text{AlK}(\text{SO}_4)_2$.
 Aluminum hydroxide. (No purity rubric.) $\text{Al}(\text{OH})_3$.
 Aluminum sulphate. At least 99.5 per cent. absolute $\text{Al}_2(\text{SO}_4)_3 + 16\text{H}_2\text{O}$.
 Kaolin. The purified ore. $\text{H}_2\text{Al}_2\text{Si}_2\text{O}_8 + \text{H}_2\text{O}$.
 Cataplasma of kaolin. A pharmaceutical.

ALUMEN. Alum.



It should contain not less than 99.5 per cent. of pure Aluminum and Potassium Sulphate.

Large, colorless, octahedral crystals, sometimes modified by cubes, or in crystalline fragments; without odor, but having a sweetish and strongly astringent taste.

Soluble in 9 parts of water at 25°C . (77°F .) and in 0.3 part of boiling water; it is also freely soluble in warm glycerin, but insoluble in alcohol.

When gradually heated it loses water; at 92°C . (197.6°F .) it fuses, and if the heat be gradually increased to 200°C . (392°F .) it loses all of its water of crystallization (45.55 per cent. of its weight), leaving a voluminous, white residue (see *Alumen Exsiccatum*).

An aqueous solution of Alum has an acid reaction upon blue litmus-paper.

Tests for Identity.—See Aluminum, Potassium, and Sulphates in Part V.

Impurities.—Ammonia alum, heavy metals, and excess of iron. Details in Part V.

Remarks.—The present pharmacopœia gives the symbolic formula of alum as $\text{AlK}(\text{SO}_4)_2 + 12\text{H}_2\text{O}$, instead of the time-honored formula $\text{K}_2\text{Al}_2(\text{SO}_4)_4 + 24\text{H}_2\text{O}$. Since the latter more clearly explains the composition of alum, we will use it in the following discussion.

At first glance, the symbolic formula, $\text{K}_2\text{Al}_2(\text{SO}_4)_4 + 24\text{H}_2\text{O}$, seems exceedingly complicated, but viewed in the light of the pharmacopœial synonym—aluminum and potassium sulphate—it becomes simplified. What is aluminum sulphate? We have just learned above that it is $\text{Al}_2(\text{SO}_4)_3$. And potassium sulphate? This is derived from sulphuric acid, H_2SO_4 ; by replacing its two hydrogen atoms with two atoms of the univalent element, potassium:

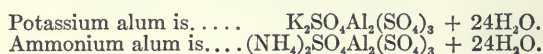
Hence,
 If we add to this aluminum sulphate, $\frac{\text{K}_2\text{SO}_4}{\text{Al}_2(\text{SO}_4)_3}$,
 We get $\text{Al}_2\text{K}_2(\text{SO}_4)_4$,

and if we crystallize this with 24 molecules of water of crystallization, we have the formula $\text{K}_2\text{Al}_2(\text{SO}_4)_4 + 24\text{H}_2\text{O}$.

The reason that this is not written $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 + 24\text{H}_2\text{O}$ is because the union between the sulphate of aluminum and potassium is stronger than a mere physical mixture. While not fully proved, we believe that there exists between the two salts a chemical relationship, somewhat akin to that between a salt and its water of crystallization. Official *alum* is not the only substance known by that name. For the official alum the cheaper ammonia alum is sometimes substituted.

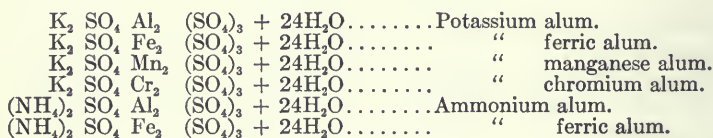
This ammonia alum was the "alumen" of the pharmacopœia of 1870, but was replaced in the pharmacopœia of 1880 by the potassa alum. A decade since ammonia alum was often furnished on orders for official alum, but at the present time it is very rarely furnished by wholesale druggists, though large quantities are still used in the arts, since cheaper than potassa alum.

Let us compare the formula of these two alums, representing them as broken into the sulphates of the two metals:



The only difference between these two is that in one we have potassium sulphate; in the other, we find ammonium sulphate, each being combined with aluminum sulphate and with 24 molecules of water of crystallization.

When we closely consider the two formulas just given, we find that each represents one molecule of the sulphate of a univalent element, one molecule of a sulphate of a trivalent element, and 24 molecules of water of crystallization. We have seen that the univalent element or radicle can be changed; for in the two alums just mentioned one contained potassium sulphate, the other ammonium sulphate. In like manner we can make a sodium alum of the formula $\text{Na}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 + 24\text{H}_2\text{O}$. We have changed the univalent element or radicle three times. Can we thus change the trivalent element? Most assuredly, replacing aluminum by the trivalent elements, iron, manganese, or chromium, getting such compounds as:



The last of these is official as ferric alum.

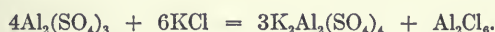
We see how many alums can be produced; hence the only general definition of an alum that can be given is that mentioned above: "the combination of one molecule of the sulphate of a univalent element or radicle, with one molecule of the sulphate of a trivalent element or radicle, and with 24 molecules of water of crystallization." Not only do all alums crystallize with the same proportion of water, but also their crystal shapes are the same—in other words, they are *isomorphous*.

Alum has been made for centuries from certain ores of aluminum, particularly alum shale. This is a form of clay containing iron

pyrites (FeS_2), and on letting the ore stand exposed to the elements for one to two years, the ferrous sulphite is changed to ferrous sulphate, ferric subsulphate, and free sulphuric acid, and the latter in turn reacts with the clay (aluminum silicate) to form aluminum sulphate. Roasting the ore after standing effects the liberation of more sulphuric acid from the iron sulphates, and the roasted mass on lixiviation yields a solution of aluminum sulphate contaminated with more or less iron. Various methods are employed in removing the iron, such as long standing of the solution exposed to the air, when the iron precipitates as ferric oxide; placing pieces of iron in the solution, which converts all the dissolved iron into ferrous sulphate, which is separated from the aluminum sulphate by fractional crystallization.

The concentrated and pure aluminum sulphate solution is then mixed with potassium chloride or sulphate, and at the proper degree of concentration crystals of alum begin to separate.

Potassium chloride is used because more abundant than potassium sulphate, and when mixed with aluminum sulphate solution, yields a double sulphate of potassium and aluminum, as shown in the following equation:



Alum is also made on a large scale by treating the aluminum hydroxide, which is a side-product in the cryolite process (p. 462), with sulphuric acid.

Alum is a valuable astringent, either internally or else externally in the form of a wash.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

ALUMEN EXSICCATUM. Exsiccated Alum.

Recipe and details of manufacture in Part VII.

Remarks.—This official is obtained by the exsiccation of crystallized alum, 100 parts of the crystal yielding 55 parts of the dried alum. (See p. 1101.)

Exsiccated alum is used as a mild escharotic under the name of *burnt alum*, being particularly efficacious for the removal of that form of morbid growth developing in an open sore called proud flesh. It is very rarely given internally.

ALUMINI HYDROXIDUM. Aluminum Hydroxide.

[ALUMINI HYDRAS, PHARM. 1890]

Recipe.—Alum, one hundred grammes..... 100 Gm.
 Monohydrated Sodium Carbonate, forty-three
 grammes..... 43 Gm.
 Water, a sufficient quantity.

Dissolve each salt separately in *one thousand cubic centimeters* of Water, filter the solutions and heat them to boiling. Then having poured the hot solution of Monohydrated Sodium Carbonate into a capacious vessel, gradually pour in the hot solution of Alum with constant stirring, and add *two thousand cubic centimeters* of boiling Water. Allow the precipitate to subside, decant the clear liquid, and pour upon the precipitate *two thousand cubic centimeters* of hot Water. Again decant, transfer the precipitate to a strainer, and wash it with hot Water until the washings produce not more than a faint cloudiness with barium chloride T. S. Then allow it

to drain, dry it at a temperature not exceeding 40° C. (104° F.), and reduce it to a uniformly fine powder.

A white, light, amorphous powder, odorless and tasteless, and permanent in dry air.

Insoluble in water or alcohol, but completely soluble in hydrochloric or sulphuric acid, and also in potassium hydroxide T.S.

When heated to redness, it loses about 34 per cent. of its weight.

If 1 Gm. of the salt be boiled with 20 Cc. of distilled water, and the liquid filtered, the filtrate should not have an alkaline reaction, and on evaporation should have a residue weighing not more than 0.005 Gm. (limit of *alkali salts*).

Tests for Identity.—Given above.

Impurities.—Excess of iron, sulphate, and heavy metals. Details in Part V. Excess of alkali salts, see above.

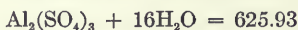
Remarks.—Aluminum hydroxide is made by adding a solution of alum to a solution of sodium carbonate. This may at first produce aluminum carbonate, but if such is the case, it is only for an instant, the carbonate decomposing with the evolution of carbon dioxide, forming a precipitate of aluminum hydroxide. In preparing aluminum hydroxide it is important that the alum solution be added to the sodium carbonate solution, and not the reverse method, as in the latter case the hydroxide will be contaminated with alkaline sulphates.

A point of importance in the manufacture of chemicals by precipitation is this very question of which chemical solution should be added to the other. The pharmacist, therefore, should carefully consult the best authorities before attempting such a precipitation, as it frequently happens that while the product yielded by one method is perfectly pure, that yielded by the reverse process is practically worthless.

One of the most interesting things in connection with aluminum hydroxide is its vast capacity for water, a fresh, well-drained magma of aluminum hydroxide containing no less than 95 per cent. of water.

Aluminum hydroxide is a valuable absorbent, and therefore useful in dusting-powders.

ALUMINI SULPHAS. Aluminum Sulphate.



It should contain not less than 99.5 per cent. of pure Aluminum Sulphate.

A white, crystalline powder, or shining plates, or crystalline fragments; without odor, having a sweetish and afterward an astringent taste, and permanent in the air.

Soluble in 1 part of water at 25° C. (77° F.), more soluble in boiling water, but insoluble in alcohol.

When gradually heated to about 200° C. (392° F.) it loses its water of crystallization (45.7 per cent. of its weight).

The aqueous solution of the salt has an acid reaction upon blue litmus-paper.

A filtered, aqueous solution of the salt (1 in 10) should not become more than faintly opalescent within five minutes after the addition of an equal volume of tenth-normal sodium thiosulphate V.S. (limit of *free acid*).

Tests for Identity.—See Aluminum and Sulphates in Part V.

Impurities.—Ammonia, heavy metals, excess of iron. Details in Part V. Excess of free acid, see above.

Remarks.—This official is prepared by dissolving moist aluminum hydroxide in diluted sulphuric acid, and granulation of the salt by evaporation of the solution.

Aluminum sulphate is used internally as an antiseptic, but chiefly externally as an astringent. Its astringent properties make it of great value as a fixer in dyeing.

KAOLINUM. Kaolin.

A native aluminum silicate, consisting chiefly of the pure silicate $[H_2Al_2Si_2O_8 + H_2O = 257.12]$, powdered and freed from gritty particles by elutriation.

A soft, white or yellowish-white powder, or in lumps, having an earthy or clay-like taste, insoluble in water, and in cold dilute solutions of the acids and alkali hydroxides.

When moistened with water, Kaolin assumes a darker color and develops a marked clay-like odor.

If 1 Gm. of Kaolin be mixed with 10 Cc. of water and 5 Cc. of sulphuric acid in a porcelain dish, no effervescence should occur, and if the mixture be evaporated until the excess of water has been removed, and further heated until dense white fumes of sulphuric acid anhydride appear, then after cooling and adding 20 Cc. of water, boiling for a few minutes and filtering, there should remain on the filter a gray insoluble residue of *impure silica*.

If Kaolin be ignited at a red heat, it should leave not less than 85 per cent. of non-volatile residue.

If to one-half of the filtrate ammonia water be added, a gelatinous precipitate of *aluminum hydroxide*, insoluble in excess, should be obtained.

If to the remaining half of the filtrate, sodium hydroxide T.S. be added, it should yield a gelatinous precipitate which is almost or completely soluble in an excess of the reagent.

If 2 Gm. of Kaolin be rubbed in a mortar with 10 Cc. of water, the mixture should not acquire more than a slight reddish tint on the addition of 0.5 Gm. of sodium salicylate (absence of more than traces of *iron*).

Tests for Identity.—Given above.

Impurities.—Excess of iron, excess of moisture, and other volatile matter. Details given above.

Remarks.—This is aluminum silicate, found largely in the Department of Allier, France, and is of great value in technic as a basis of porcelain. A description of the manufacture of porcelain is beyond the limits of this work, and for this the reader is referred to any of the leading works on chemical technology mentioned in the Preface. Kaolin is of pharmaceutical importance as having been suggested for use in the manufacture of the pills of potassium permanganate and other explosive substances, these substances being mixed with kaolin and made into a mass by the addition of soft petrolatum. Kaolin is now largely used in the form of poultice, made by triturating the finely powdered clay with glycerin to a thick paste. This is recognized in the present pharmacopœia as Cataplasma of Kaolin. (See p. 352.)

CERIUM

Symbol, Ce. Atomic weight, 139.2

Cerium is a rare metal, similar in many respects to aluminum. It is of technical importance because the "mantle" of the Welsbach burner—that well-known mode of illumination—is made of a network of oxides of cerium, thorium, didymium, and lanthanum, the last two elements usually accompanying cerium in its ores.

The sole pharmaceutical interest in cerium is because one of its salts, the oxalate, is official.

CERII OXALAS. Cerium Oxalate.

Cerium Oxalate consists chiefly of a mixture of the oxalates of cerium, didymium, and lanthanum, and of other rare earths of this group.

A fine, white powder, without odor or taste, and permanent in the air.

Insoluble in water, alcohol, ether, or in solutions of potassium or sodium hydroxide; insoluble in cold, but soluble in hot, diluted sulphuric or hydrochloric acid.

When heated to redness it is decomposed, leaving a residue of reddish-brown ceric and other rare-earth oxides, constituting not less than 47 per cent. of the salt.

On boiling the salt with potassium hydroxide T.S., an insoluble residue of white hydroxides is produced; if the filtrate from this residue be supersaturated with acetic acid, the addition of calcium chloride T.S. will produce a white precipitate, insoluble in acetic acid, but soluble in hydrochloric acid.

If the residue left after heating Cerium Oxalate be dissolved in concentrated sulphuric acid, and a small crystal of strychnine added, a deep blue color will appear, which will rapidly change to purple and then to red.

From the solution in diluted hydrochloric or sulphuric acid, potassium hydroxide T.S. precipitates white hydroxides, which do not redissolve in an excess of the reagent, and gradually turn yellow in contact with air. Ammonium carbonate T.S. precipitates from the same solution white cerous and other rare-earth carbonates, which are somewhat soluble in an excess of the reagent.

If 0.1 Gm. of Cerium Oxalate be dissolved in 1 Cc. of sulphuric acid, and 2 Cc. of potassium sulphate T.S. be added, small, colorless crystals of cerium and other rare-earth potassium sulphates will, after some time, be deposited.

Tests for Identity.—Given above.

Impurities.—Carbonate, heavy metals, arsenic, aluminum, and zinc. Details in Part V.

Remarks.—Cerium oxalate is made as a by-product of thorium salts from the ore, *monazite*, the thorium salts being in great demand for making Welsbach mantles. Cerium oxalate was formerly made from the mineral *cerite* by a process necessarily complicated, in order to free it, as much as possible, from lanthanum and didymium, two closely allied rare metals invariably accompanying cerium and its ores.

It will be seen, from the official definition given above, that a complete separation of these rare metals is impossible, and the oxalates thereof are permitted in the official salt.

It is a valuable remedy in the vomiting of pregnancy.

Dose.—65 milligrammes (1 grain).

CHROMIUM

Symbol, Cr. Atomic weight, approximately 52.

Chromium, like manganese, aluminum, zinc, and iron, stands midway between the positive and negative elements, forming chromium salts, such as chromous sulphate, CrSO_4 , with strong acids; and chromates, such as potassium chromate, K_2CrO_4 , with alkalis.

However, it is usually more negative than positive, and in its two official compounds, chromium trioxide and potassium dichromate, it is strongly negative.

The chief ore of chromium is chrome iron ore, FeOCr_2O_3 . (See p. 446.)

The word chromium is derived from the Greek word *chromos*, meaning color, and is so called by reason of the brilliant characteristic color of the various chromium compounds. This name was bestowed on the element by its discoverer, Vauquelin.

The element chromium is isolated either by the reduction of

chromic oxide, with soot or coke, or, better, by fusion of a mixture of the chlorides of chromium, potassium, and sodium with zinc. The alkaline chlorides act as a flux, while an alloy of zinc and chromium deposits in the bottom of the crucible. This alloy is treated with diluted nitric acid, which dissolves the zinc, leaving chromium as a gray powder.

Chromium is a gray-white, brittle metal, hard enough to scratch glass, and more difficult to melt than platinum. It is not easily oxidized by contact with air.

The characteristic tests of chromium are as follows:

Ammonium sulphide precipitates, from the solution of chromium compounds, green chromic hydroxide, potassium hydroxide producing the same precipitate; lead acetate solution precipitates solutions of chromium compounds in the form of a yellow lead chromate (chrome yellow), which is rather largely esteemed as a paint. It should not, however, be used in coloring buns in place of the yolk of egg, as was tried by the bakers of Philadelphia some twenty years ago, with disastrous results.

Chromium yields several oxides—chromium oxide, CrO ; chromium tetroxide, Cr_3O_4 ; chromic oxide, Cr_2O_3 ; chromium dioxide, CrO_2 ; chromium trioxide, CrO_3 . The variation in valence represented by these oxides leads to considerable confusion, and for this reason the element chromium was omitted from the table of elements on p. 367. A similar confusion is encountered in the naming of its oxides, it being particularly unfortunate that the term chromic oxide is applied to Cr_2O_3 . We have been taught that an "ic" oxide should yield the corresponding "ic" acid, and yet the official formerly called "chromic acid" is not derived from Cr_2O_3 , but is none other than chromic trioxide, mentioned above.

CHROMII TRIOXIDUM. Chromium Trioxide.



[ACIDUM CHROMICUM, PHARM. 1890]

It should contain not less than 90 per cent. of pure Chromium Trioxide (chromic acid anhydride). It should be kept in glass-stoppered bottles, and great caution should be observed to avoid bringing it in contact with organic substances, such as cork, tannic acid, sugar, alcohol, collodion, etc., as serious accidents are liable to result.

Small, needle-shaped crystals or rhombic prisms, of a dark purplish-red color and metallic lustre; odorless; destructive to animal and vegetable tissues; deliquescent in moist air.

Very soluble in water, forming an orange-red solution. When brought in contact with alcohol, ether, glycerin, and other organic solvents, decomposition takes place, sometimes with dangerous violence.

When Chromium Trioxide is heated, its color darkens, and finally becomes black, but it is restored on cooling. At 192° to 193°C . (377.6° to 379.4°F .) it fuses to a reddish-brown liquid, which, on cooling, forms a dark red, brittle mass (often enclosing cavities filled with crystals), furnishing a scarlet powder. Above 250°C . (482°F .) it begins to decompose into green chromic oxide and free oxygen, and, after protracted heating, leaves a residue of pure chromic oxide, which should yield nothing soluble in water.

When warmed with hydrochloric acid, chlorine is evolved.

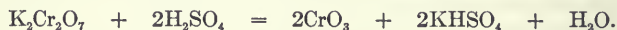
Tests for Identity.—Given above.

Impurities.—Sulphuric acid. Details in Part V.

Remarks.—This compound has the formula CrO_3 . In this, as in sulphuric oxide, SO_3 , the valence of the characteristic element is vi. It will be seen that this so-called acid is in reality an oxide, the true acid (the formula of which is H_2CrO_4 , and thus similar to that of sulphuric acid, H_2SO_4) existing only in solution.

The official is, therefore, the anhydride of true chromic acid; an anhydride of an acid being an acid deprived of what water it possessed in the form of hydroxyl groups. This is the first acid anhydride which we have studied that is commonly given the name of the acid; the other official one being arsenous acid. In both cases they are called acids instead of oxides because they are the nearest approach to the true acid which can be obtained in the solid form, and, moreover, on dissolving these oxides in water, the true acid is produced.

Chromic acid is made by treating the potassium dichromate with a molecular quantity of sulphuric acid by the following reaction:



It will be seen, from the equation, that only enough sulphuric acid is used to convert the potassium into potassium bisulphate. This salt is removed by crystallization, and the mother liquid then further concentrated, when the crop of chromic acid crystals is produced. These crystals cannot be dried as in ordinary cases by placing on bibulous paper, since chromium trioxide is a very powerful oxidizing agent, and will decompose most organic substances, particularly paper. Therefore, instead of paper, some inorganic absorbing surface must be used. For this purpose unburnt porcelain or clean fire-brick is excellent. If the use of either of these is inconvenient, drying plates of plaster of Paris can be used with excellent results. Such plates are made by making a fairly thick paste of plaster of Paris in water, and allowing same to set in an appropriate mold, for example, the cover of a pasteboard box—and after thoroughly drying the porous plate is obtained.

By reason of the oxidizing action of the chromic acid the pharmacopœia sounds a note of warning against triturating this chemical with easily oxidizable material. Hence beware of prescriptions directing chromic acid dispensed in pills. These must not be made with ordinary excipient, but with petroleum mass (kaolin and soft petrolatum). The oxidizing action of chromic acid is used for the cleaning of mortars, as already explained on p. 114. Chromic acid is also very largely used in oxidization processes in organic chemistry, for such purposes where nitric acid would act too powerfully. Medicinally, chromic acid is used almost entirely as a caustic, and its use in this way is usually limited to the removal of corns or warts. For this purpose it is very effective, but it should be applied only by a skilled operator, as careless handling of the chemical in such cases is apt to result in dangerous wounds.

POTASSII DICHROMAS. Potassium Dichromate.

This salt has already been discussed under the potassium compounds, and here it is only necessary to state that it is used chiefly as an oxidizing agent, and for this purpose is used with sulphuric acid, thus liberating chromic acid.

CHAPTER XXXI

IRON

Symbol, Fe. Atomic weight, approximately 56

THE most useful and abundant of metals is *iron*. Its use is so diversified, both technically and pharmaceutically, that we must give it careful consideration. No other metal furnishes more officials, and almost without exception these are valuable.

The element is widely distributed on the earth, but in no place as the pure metal. It oxidizes (rusts) so rapidly that it cannot long remain pure in the earth's atmosphere. We find pure native iron only in meteoric stones which fall from the heavens, and are supposedly fragments of the sun's substance. This leads us to the conclusion that the sun's atmosphere must be composed of some gases other than oxygen.

The principal ores of iron found on the earth's surface are as follows: *Magnetic iron ore* or *lode-stone*. This ore, the history of which has been discussed on p. 527, has the formula Fe_3O_4 , and is supposed to be a ferrous ferrate, $\text{Fe}(\text{FeO}_2)_2$, being derived from a hypothetical acid, HFeO_2 , which is analogous to the aluminic acid already described on p. 463.

The name ferric acid is not applied to this compound, however, as it appears that there is an acid, H_2FeO_4 , in which iron shows the valence vi.

The potassium salt of this acid, K_2FeO_4 , is known.

This magnetic iron ore is used more by reason of its magnetic properties than as a source of iron. The commercial ores are chiefly *red hematite*, Fe_2O_3 , *spathic iron ore*, FeCO_3 , and *iron pyrites*, FeS_2 . This last has already been discussed on p. 422, as one of the sources of sulphuric acid, so here it need only be stated that the term pyrites is derived from the Greek word *pyros*, fire, and is used in this case because, when the ore is struck, it emits sparks. Iron pyrites is also interesting as being the only iron compound in which iron shows the valence iv. Besides these commercial sources of iron, reference should be made to the fact that this element is found in chlorophyll and in the blood, and in both cases its presence is necessary to the life of both plant and animal. So soon as the chlorophyll of the plant or the hematin of the blood becomes deficient in iron, the assimilation processes of the plant or animal economy lessens, with corresponding disastrous results to the organism. When iron is absent from the chlorophyll plants or from the blood corpuscles, such plants or animals are said to be anemic.

Iron has been known from ancient times as one of the most valuable of metals. In fact, one of the most important steps in the development and progress of humanity is known by the anthropologists as the transition from the stone to the iron age.

VARIETIES OF IRON

The commercial forms of the metal iron can be roughly divided into three groups—cast iron, steel, and wrought iron. The difference

between these three groups is based on the amount of carbon contained in the metal. Thus, *cast iron* contains from 3 to 6 per cent. carbon; *steel*, from 0.8 to 1.8 per cent. of carbon; and *wrought iron*, from 0.2 to 0.6 per cent.

The origin of these different varieties of iron is merely a question of the manipulation. Cast iron is prepared by treating the ore (an oxide of iron) with coal and limestone in the presence of a blast of air. The coal in this case acts as a reducing agent, as shown in the following equation:



The carbon monoxide produced also acts as a reducing agent on the ore, ultimately escaping as carbon dioxide (CO_2). The function of the limestone is to form a combination with the silicates naturally found in ore, the resulting calcium silicate separating in the form of *slag*, it floating on the surface of the molten iron. This process goes on in a special apparatus (blast furnace) lined with brick or fire-clay, and it is so arranged that the process is continuous. Alternative layers of the ore, coal, and limestone being dumped in at the top, meeting the fiery blast produced by combustion of the preceding portions of coal about midway down in the apparatus, and molten iron drawn from the bottom.

Wrought iron is prepared by removing all the carbon from the cast iron by keeping the latter in the molten state for a time sufficient to burn the carbon contained therein by contact with the air. There are two general methods of accomplishing this result: first and oldest is called puddling, and consists of melting the cast iron in flat vessels having considerable surface exposed to the air, and stirring same vigorously with an appropriate paddle or spoon until all the carbon has combined with the oxygen of the air. In the more modern method the molten cast iron is submitted to the action of an air-blast, being constantly stirred all the while.

Steel, as will be noted above, contains a carbon percentage midway between that of cast iron and wrought iron. Hence, by the proper blending of these two forms of iron, a steel of any carbon percentage can be obtained, the calculation in this case being by the process of alligation, similar to that already described on p. 59.

This blending is performed by the Bessemer process, the operation being carried on in a special apparatus called the Bessemer converter, consisting of a huge, pear-shaped vessel, lined with fire-brick, revolving on a pivot in such a way that it can be inverted. Into this converter the molten cast iron is poured, a blast of air passed therein until the cast iron is converted into wrought iron. The wrought iron is then blended with sufficient cast iron to make a finished product of exactly the carbon content desired. It is needless to say that this explanation of the manufacture of the three forms of iron is superficial, extended comment thereon being beyond the limits of this volume, and for full details of the process the reader is referred to the books on chemical technology, given in the Bibliography in the Preface.

It might, however, be stated that, for the modern manufacture of steel, many recipes are employed, and each modification is usually the secret of the individual ironmaster. Thus, in making these special

steels many of the rarer elements, such as uranium, are added to impart the desired toughness to the steel.

One modification of steel manufacture is worthy of passing notice. A great drawback in the manufacture of steel from the iron ore obtained in continental Europe was due to the fact that this ore contained a considerable amount of phosphorus, which caused the finished steel to be brittle, and hence comparatively useless. A remedy for this condition was discovered by Thomas and Gilchrist, who employed the simple method of lining the Bessemer converter with calcined dolomite. When the phosphorus-bearing ores were converted into wrought iron in such converters, the phosphorus is absorbed by the calcium and magnesium of the dolomite lining, with the formation of the phosphates of these two metals. Not only does this process afford a ready means of removing the phosphorus from the ore, but the dolomite lining is thereby converted into calcium and magnesium phosphates, having ready sale as a fertilizer.

The properties of iron must be discussed under the three diversified forms just described. All three forms of iron—cast iron, steel, and wrought iron—act chemically as iron, all dissolving in acids to form ferrous, and eventually ferric, salts. Another point showing their iron origin is the fact that all, when exposed to the moist atmosphere, oxidize with the formation of Fe_2O_3 , which is familiar to all under the name of *iron-rust*. Physically, the three forms show a marked similarity. *Cast iron*, for example, melts at 1150°C. , is quite brittle, and hence breaks when hammered. This variety is called cast iron because it shares with water the property of expanding at the moment of solidification—a property not found in any other metal. *Steel* melts at 1400°C. , and is the reverse of brittle, being, when properly tempered, the most elastic of metals. It is very hard, and hence it is but slightly affected by the blows of a hammer, not breaking as cast iron does, and not flattening, as does wrought iron. *Wrought iron* melts at 1500°C. , and is neither brittle nor elastic, but is very malleable. In fact, a commercial term for this form of iron is *malleable iron*, and to this property is due its use in the arts.

Of the three commercial forms of iron just mentioned, wrought iron is the nearest approach to the pure element. The other two, steel and cast iron, being now considered as mixtures of iron with varying proportions of iron carbide.

Tests.—The tests for iron and its salts are as follows: The black precipitate obtained by treating its solution with ammonia sulphide; the reddish-brown magma by treatment of ferric solutions with ammonium hydroxide, this precipitate being official under the name of ferri oxidum hydratum; and the black coloration produced on treating an iron solution with tannin. This latter mixture, when combined with appropriate adhesive agents, forms the typical *black ink*, and is of pharmaceutical interest because such black coloration is produced whenever an iron salt is combined with the preparation of a drug containing tannin.

Besides these tests, far more characteristic are potassium ferricyanide, which gives a blue precipitate with ferrous salts, and potassium ferrocyanide, which gives a blue precipitate with ferric salts. An exceedingly delicate test for ferric salts is the red color produced by potassium sulphocyanate.

Metallic iron is recognized by the pharmacopœia in two forms, namely, *ferrum* and *ferrum reductum*.

FERRUM. Iron.

Fe = 55.5

Metallic iron, in the form of fine, bright, and non-elastic wire.

Remarks.—This is wrought iron, usually in the form of fine, bright, non-elastic wire, and a popular variety of this is what is commercially termed card-teeth, consisting of small points of iron wire used as the teeth of appliances employed in carding wool and cotton.

In dissolving card-teeth in acids, hydrogen is evolved, as shown in the following equation:



but at the same time it will be noticed that a peculiar odor is emitted. This odor is due to the hydrocarbon produced by the combination of the hydrogen with the carbon contained in the card-teeth.

Iron wire is recognized by the pharmacopœia only as a source of iron compounds, it being the most convenient form from which to make the salts of the metal.

FERRUM REDUCTUM. Reduced Iron.

Reduced Iron should contain not less than 90 per cent. of pure metallic iron.

A very fine, grayish-black, lustreless powder, without odor or taste; permanent in dry air.

Insoluble in water or alcohol.

One Gm. of Reduced Iron, when treated with 20 Cc. of diluted sulphuric acid, causes the evolution of nearly odorless hydrogen gas, which should not effect, within five minutes, paper moistened with lead acetate T.S. (limit of *sulphide*), and on applying a gentle heat the Iron should dissolve in the acid without leaving more than 1 per cent. of residue.

When ignited in contact with air, it glows and is converted into black ferrous-ferric oxide.

If 1 Gm. of Reduced Iron be shaken with 5 Cc. of water, the liquid should not change the color of red litmus-paper.

Tests for Identity.—Given above.

Impurities.—Arsenic. Details in Part V.

Assay.—Details in Part V.

Reduced iron represents a chemically pure form of iron; hence supposedly entirely free from carbon. It is prepared by placing subcarbonate of iron (virtually, ferric oxide, Fe_2O_3) in a tube of Bohemian glass or a piece of iron piping, some four feet long, and one-half to one inch in diameter. Hydrogen, produced in the ordinary way (zinc and acid, see p. 519), is passed through the tube, which is placed in an appropriate coal or gas furnace and heat is applied after the air has been expelled from the tube by the current of hydrogen, which, passing over the heated ferric oxide, reduces it to pure iron by the following reaction:



This explains its synonym, *iron by hydrogen*.

Before removing the reduced iron from the tube, care must be taken to see that the same be perfectly cooled. If the iron be poured

out while still hot, its affinity for the oxygen of the air is so great that it directly combines therewith, with the formation of a vivid flame. Such substances which spontaneously ignite when brought into contact with the air are said to be *pyrophoric*, and as a striking illustration may be cited antimony sulphide. Some writers claim that pure reduced iron is not pyrophoric, that phenomena being caused by presence of ferrous oxide, FeO .

Reduced iron is a valuable tonic.

Dose.—65 milligrammes (1 grain).

The following compounds of iron are official:

Ferrous Salts:

Saccharated ferrous carbonate. At least 15 per cent. absolute FeCO_3 .

Mass of ferrous carbonate contains about 40 per cent. FeCO_3 .

Pills of ferrous carbonate contain 0.16 Gm. ferrous sulphate and 0.08 Gm. potassium carbonate to each pill.

Mixture of ferrous carbonate contains about $\frac{1}{4}$ of 1 per cent. FeCO_3 .

Syrup of ferrous iodide. At least 5 per cent. absolute FeI_2 .

Pills of ferrous iodide contain 0.04 Gm. iron and 0.05 Gm. iodine to each pill.

Ferrous sulphate. At least 99.5 per cent. absolute $\text{FeSO}_4 + 7\text{H}_2\text{O}$.

Exsiccated ferrous sulphate. Each 65 Gm. represent 100 Gm. $\text{FeSO}_4 + 7\text{H}_2\text{O}$.

Granulated ferrous sulphate. $\text{FeSO}_4 + 7\text{H}_2\text{O}$.

Ferric Salts:

Ferric chloride (FeCl_3) contains at least 22 per cent. metallic iron.

Solution of ferric chloride contains 29 per cent. absolute FeCl_3 or 10 per cent. metallic iron.

Tincture of ferric chloride contains 13.28 per cent. absolute FeCl_3 or 4.58 per cent. metallic iron.

Ferric ammonium sulphate. At least 99.5 per cent. absolute $\text{FeNH}_4(\text{SO}_4)_2 + 12\text{H}_2\text{O}$.

Ferric hydroxide. A magma consisting of $\text{Fe}(\text{OH})_3$ and water.

Ferric hydroxide with magnesium oxide. A magma consisting of $\text{Fe}(\text{OH})_3$, magnesium hydroxide, and water.

Ferric hypophosphite. At least 98 per cent. absolute $\text{Fe}(\text{PH}_2\text{O}_2)_3$.

Solution of ferric sulphate contains about 36 per cent. $\text{Fe}_2(\text{SO}_4)_3$ or 10 per cent. metallic iron.

Solution of ferric subsulphate consists of basic ferric sulphate; contains 13.57 per cent. metallic iron.

Solution of iron and ammonium acetate. A pharmaceutical containing 4 per cent. tincture of ferric chloride.

Scale Salts of Iron:

Ferric citrate contains at least 16 per cent. metallic iron.

Iron and ammonium citrate contain at least 16 per cent. metallic iron.

Wine of iron contains 4 per cent. iron and ammonium citrate.

Iron and quinine citrate contain at least 13 per cent. metallic iron.

Soluble iron and quinine citrate contain at least 13.5 per cent. metallic iron.

Bitter wine of iron contains 5 per cent. soluble iron and quinine citrate.

Iron and strychnine citrate contain at least 16 per cent. metallic iron.

Soluble ferric phosphate contains at least 12 per cent. metallic iron.

Glycerite of the phosphates of iron, quinine, and strychnine contains 8 per cent. soluble ferric phosphate.

Syrup of the phosphates of iron, quinine, and strychnine contains 25 per cent. of the above glycerite.

Elixir of iron, quinine, and strychnine phosphates contains $1\frac{1}{2}$ per cent. of soluble ferric phosphate.

Soluble ferric pyrophosphate contains at least 10 per cent. metallic iron.

Iron and ammonium tartrate contains at least 13 per cent. metallic iron.

Iron and potassium tartrate contains at least 15 per cent. metallic iron.

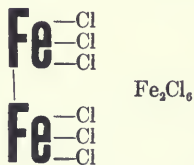
It will be seen that iron compounds in the above table are grouped into ferrous salts, ferric salts, and scale salts of iron. The latter, which are pharmaceutic preparations rather than definite chemicals, will be discussed by themselves (p. 566). The ferrous salts and ferric salts are combinations of acids with iron, the latter exhibiting two different valences.

Reference to the elemental table on p. 367 shows that iron is one of the positive elements which possess more than one valence, and that, while the variation in the valence of negative elements is almost invariably a difference of two,—say I, III, V, VII, or II, IV, VI,—thus agreeing with Frankland's bond idea, explained on p. 364, the variation of iron, expressed in that table, is II and III, the two phases of the element being expressed as ferrous and ferric respectively.

The formulas of ferrous compounds, such as ferrous iodide, FeI_2 , or ferrous sulphate, FeSO_4 , is easy to comprehend if one bears in mind that bivalent iron—iron with two free bonds—acts exactly as does bivalent calcium, and all that was said of the formulas of alkaline earth metals on p. 495 applies to ferrous salts.

In giving the valence III to the ferric form clearness is possibly sacrificed for brevity, and it is well to explain that there has been much discussion on the valence of ferric iron, some giving the valence III (Fe^{III}), and others giving two molecules of iron with the valence VI—(Fe_2)^{VI}. The latter view was that accepted by the United States Pharmacopœia of 1890, but at the last revision the trivalent view was accepted, and as a result the formula of ferric chloride is given as FeCl_3 in the present pharmacopœia.

The formula Fe_2Cl_6 is accepted by some chemists (Deville and Troust: also Friedel and Crafts) because, in the first place, the molecular weight estimations place it nearer that of the formula Fe_2Cl_6 than that of FeCl_3 , and then, too, if the formula Fe_2Cl_6 prevails, it would show the valence of ferric iron to be IV, thus maintaining the regularity in valence variation noticeable in the negative elements. If iron has the valence IV, two atoms would link together just as two carbon atoms link in ethane (see p. 627), giving the following formula:



The only reason that such formulas as FeCl_3 are employed instead of Fe_2Cl_6 is for sake of brevity.

FERROUS SALTS

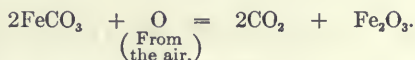
Ferrous carbonate, FeCO_3 , is official in four forms: the saccharated, the mass, the pills, and compound iron mixture. In all these preparations the carbonate is manufactured in about the same way—treatment of ferrous sulphate with an alkaline carbonate. In the saccharated, sodium bicarbonate is used; in the mass, sodium carbonate;

and in the other two, potassium carbonate is employed, the reaction in the latter case being—



while, of course, in the other the reaction is quite analogous.

Uncombined ferrous carbonate is very unstable, going over into ferrous oxide by the following reaction:



The iron carbonate or subcarbonate of iron usually found in stores, a red-brown powder, is almost entirely Fe_2O_3 —common iron-rust. The oxidation of the carbonate may be partly prevented by protecting the salt with sugar, and accordingly we find that all four official preparations just mentioned have sugar as an ingredient.

FERRI CARBONAS SACCHARATUS. Saccharated Ferrous Carbonate.

Saccharated Ferrous Carbonate should contain not less than 15 per cent. of Ferrous Carbonate [$\text{FeCO}_3=115.05$] and should be kept in small, well-stoppered bottles.

<i>Recipe.</i> —Ferrous Sulphate, <i>fifty grammes</i>	50 Gm.
Sodium Bicarbonate, <i>thirty-five grammes</i>	35 Gm.
Sugar, <i>in fine powder</i> ,	
Distilled Water, <i>each, a sufficient quantity</i> ,	
To make <i>one hundred grammes</i> ..	100 Gm.

Dissolve the Ferrous Sulphate in *two hundred cubic centimeters* of hot Distilled Water, and the Sodium Bicarbonate in *five hundred cubic centimeters* of Distilled Water at a temperature not exceeding 50°C . (122°F .), and filter the solutions separately. To the solution of Sodium Bicarbonate contained in a flask having a capacity of about *one thousand cubic centimeters* add, gradually, the solution of Ferrous Sulphate, and mix thoroughly by rotating the flask. Fill the flask with boiling Distilled Water, cork it loosely, and set the mixture aside. When the precipitate has subsided, draw off the clear supernatant liquid by means of a siphon, and then fill the flask again with hot Distilled Water, and shake it. Again draw off the clear liquid, and repeat the washing with hot Distilled Water in the same manner until the decanted liquid gives merely a slight cloudiness with barium chloride T.S. Finally, drain the precipitate thoroughly on a muslin strainer, transfer it to a porcelain dish containing *eighty grammes* of Sugar, and mix it intimately. Evaporate the mixture to dryness, by means of a water-bath, reduce it to powder, and mix intimately with it, if necessary, enough well-dried Sugar to make the final product weigh *one hundred grammes*.

A greenish-brown powder, gradually becoming oxidized by contact with air, without odor, and having at first a sweetish, afterward a slightly ferruginous taste.

Only partially soluble in water, but completely soluble upon the addition of hydrochloric acid, with copious evolution of carbon dioxide, forming a clear, greenish-yellow liquid.

Tests for Identity.—Given above.

Impurity.—Sulphate. Details in Part V.

Assay.—Details in Part V.

It will be seen, from the above process, that this compound is made by pouring a solution of ferrous sulphate into a solution of sodium bicarbonate, and washing the precipitated ferrous carbonate with boil-

ing water, all the time using precautions to prevent access of air. The washed precipitate is then mixed with sugar, and evaporated to dryness on a water-bath, and finally mixed with a sufficient quantity of sugar to make the finished product contain about 15 per cent. of ferrous carbonate.

The saccharated carbonate has all the properties of ferrous carbonate, and is a chalybeate tonic, particularly indicated in anemia.

Dose.—250 milligrammes (4 grains).

MASSA FERRI CARBONATIS. Mass of Ferrous Carbonate.

The recipe for this body, commonly called *Vallet's mass*, as well as full details of manufacture, will be found in Part VII.

It is made by pouring a solution of ferrous sulphate into a solution of sodium carbonate, removing the resulting sodium sulphate by washing with warm water, to which a sufficient amount of syrup has been added to prevent oxidation; the drained product is then combined with honey and syrup. This preparation should contain 36 to 42 per cent. of ferrous carbonate, as is figured out on p. 1128.

Dose.—250 milligrammes (4 grains).

PILULÆ FERRI CARBONATIS. Pills of Ferrous Carbonate.

<i>Recipe.</i> —Granulated Ferrous Sulphate, sixteen grammes..	16 Gm.
Potassium Carbonate, eight grammes.....	8 Gm.
Sugar, four grammes.....	4 Gm.
Tragacanth, in fine powder, one gramme.....	1 Gm.
Althæa, in No. 60 powder, one gramme.....	1 Gm.
Glycerin,	
Water, each, a sufficient quantity,	
To make one hundred pills..	100

Rub the Potassium Carbonate, in a mortar, with a sufficient quantity (about *ten drops* each) of Glycerin and Water, then add the Ferrous Sulphate and Sugar, previously triturated together to a uniform powder, and rub the mass thoroughly, until it assumes a greenish color. When the reaction has terminated, incorporate the Tragacanth and Althæa, and, if necessary, add a little more Water, so as to obtain a mass of pilular consistence. Divide this into *one hundred pills*.

These pills should be freshly prepared when wanted.

It will be seen that these pills, which are commonly called *Blaud's pills*, are made by treating potassium carbonate with ferrous sulphate and sugar, glycerin, and water, and finally incorporating althæa and tragacanth as excipients. The mass is cut and rolled into pills. In making these pills it would be well if no reaction took place between the potassium carbonate and ferrous sulphate until the pill had dissolved in the juices of the stomach.

By directing that the mass be beaten thoroughly until it assumes a greenish color, the pharmacopœia defeats this purpose and affords a product distinctly inferior to Vallet's mass. From the latter the alkaline sulphate has been removed, and the ferrous carbonate is protected by the use of sugar and honey; whereas, the pills, of which we are speaking, still contain the potassium sulphate and a lesser amount of protecting sugar. Efforts to make Blaud's pills in which the reaction has not taken place has resulted in many ingenious devices: for instance—one firm furnishes a gelatin-coated pill in which the

ferrous sulphate is placed in one-half and the potassium carbonate in the other half, being separated in the gelatin membrane. The retail pharmacist can easily prepare a similar form of pill, if he so desires, by placing the constituents, one on top of the other, in a gelatin capsule, the two chemicals being separated by a layer of sugar of milk.

Dose.—2 pills.

MISTURA FERRI COMPOSITA. Compound Iron Mixture.

<i>Recipe.</i> —Ferrous Sulphate, in clear crystals, <i>six grammes</i> ..	6 Gm.
Myrrh, in small pieces, <i>eighteen grammes</i>	18 Gm.
Sugar, <i>eighteen grammes</i>	18 Gm.
Potassium Carbonate, <i>eight grammes</i>	8 Gm.
Spirit of Lavender, <i>sixty cubic centimeters</i>	60 Cc.
Rose Water, <i>a sufficient quantity</i> ,	
To make <i>one thousand cubic centimeters</i> ..	1000 Cc.

Rub the Myrrh, Sugar, and Potassium Carbonate in a mortar, with *seven hundred cubic centimeters* of Rose Water, at first very gradually added, so that a uniform mixture may result. Transfer this to a graduated vessel, add the Spirit of Lavender, then the Ferrous Sulphate, previously dissolved in about *fifty cubic centimeters* of Rose Water, and, lastly, enough Rose Water to make the product measure *one thousand cubic centimeters*. Mix the whole thoroughly. This preparation should be freshly made when wanted.

Griffith's mixture is made by placing a mixture of myrrh, sugar, potassium carbonate, and rose water into a dispensing bottle, adding thereto spirit of lavender, and lastly a solution of ferrous sulphate in rose water.

This preparation should be freshly prepared in the bottle in which it is to be sent out. When the ferrous sulphate is added to the potassium carbonate, an evolution of carbon dioxide usually occurs, and care must be taken to permit the complete escape of this gas before the bottle be corked, otherwise there is danger of the fracture of the flask. The preparation, when fresh, is a bluish-green mixture, but on standing, especially when a few doses have been taken from the bottle, it changes to a brick-dust red—into the so-called subcarbonate of iron. The instability of this preparation makes its use, at its best, somewhat questionable, and in dispensing it the pharmacist should mention to the purchaser the color change which is bound to occur.

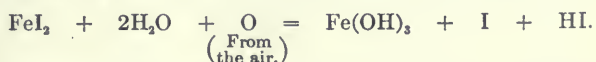
Dose.—16 Cc. (4 fluidrachms).

Ferrous iodide, FeI_2 , is another of the unstable salts of iron, to preserve which it is necessary to add sugar or some other substance capable of preventing oxidation by the atmosphere.

The reaction of manufacture is very simple: $\text{Fe} + \text{I}_2 = \text{FeI}_2$.

The salt is official in three different forms: The *saccharated*, the *syrup*, and the *pills*, which are coated with the balsam of tolu to prevent atmospheric action.

As is the case with ferrous carbonate, ferrous iodide is very unstable, splitting, when coming in contact with air, into ferric hydroxide, iodine, and hydriodic acid, as shown in the following equation:



This decomposition is largely obviated by combining ferrous iodide with sugar or hypophosphorous acid.

Ferri Iodidum Saccharatum (U. S. P. 1890) was made by preparing a solution of ferrous iodide by treatment of iodine with iron wire, mixing the filtered solution with sugar of milk, evaporating to a dry mass, and then mixing with sufficient sugar of milk to make a product containing 20 per cent. of ferrous iodide.

This product was very unstable, and hence was wisely omitted at the last revision.

SYRUPUS FERRI IODIDI. Syrup of Ferrous Iodide.

A syrupy liquid containing about 5 per cent., by weight, of Ferrous Iodide [FeI_2 , = 307.30], or about 6.74 Gm. in 100 Cc.

A transparent, pale green liquid, having a sweet, strongly ferruginous taste and a neutral reaction.

Specific gravity: about 1.349 at 25° C. (77° F.).

On adding a few drops of potassium ferricyanide T.S. to a small portion of the diluted Syrup a blue precipitate will be produced.

If mixed with a little starch T.S., and afterward with a few drops of chlorine water, the syrup will acquire a deep blue color. This color should not be produced in the Syrup by starch T.S. alone (absence of *free iodine*).

Recipe and full details of manufacture in Part VII.

Tests for Identity.—See above.

Impurity.—Iodine, see above.

Assay.—Details in Part V.

Remarks.—This syrup is made by treating iron with iodine, filtering the solution of ferrous iodide into sugar, which is dissolved therein with heat. The finished syrup is preserved by addition of a small amount of hypophosphorous acid. The syrup of the present pharmacopœia contains 5 per cent. ferrous iodide, while that of the U. S. P. 1890 was 10 per cent.

The color of spoiled ferrous iodide can be changed from the brown to the bright green by the addition of a small quantity of card-teeth or reduced iron, this seeming to show that the brown coloration is due to the presence of free iodine, rather than caramel, as has been suggested by one writer. While the problem is still undecided, it behooves the careful pharmacist never to dispense a syrup that has turned brown.

The syrup of ferrous iodide is given as an alterative and chalybeate tonic, diluted with water, and always taken through a tube, as the ferrous iodide attacks the teeth.

Dose.—1 Cc. (15 minims).

Liquor Ferri Iodidi (N. F.) is an 81 per cent. solution of ferrous iodide, preserved by addition of 2½ per cent. diluted hypophosphorous acid. One volume of this solution with 11 volumes of syrup (U. S. P.) makes a product similar to the official syrup just described.

PILULÆ FERRI IODIDI. Pills of Ferrous Iodide.

<i>Recipe</i> .—Reduced iron, four grammes.....	4 Gm.
Iodine, five grammes.....	5 Gm.
Glycyrrhiza, in No. 60 powder, four grammes.....	4 Gm.
Sugar, in fine powder, four grammes.....	4 Gm.
Extract of Glycyrrhiza, in fine powder, one gramme	1 Gm.
Acacia, in fine powder, one gramme.....	1 Gm.
Water,	
Balsam of Tolu,	
Ether, each, a sufficient quantity,	

To make one hundred pills. . 100

To the Reduced Iron, contained in a small mortar, add *six cubic centimeters* of Water, and then, gradually, the Iodine with constant stirring, until the liquid ceases to have a reddish tint. Then add the remaining powders, previously well triturated together, and mix the whole thoroughly. Transfer the mass to a porcelain dish, and evaporate the excess of moisture, on a water-bath, with constant stirring, until the mass has acquired a pilular consistence. Then divide it into *one hundred pills*.

Dissolve *ten grammes* of Balsam of Tolu in *fifteen cubic centimeters* of Ether, shake the pills with a sufficient quantity of this solution until they are uniformly coated, and put them on a plate to dry, occasionally rolling them about until the drying is completed.

Keep the pills in a well-stoppered bottle.

Pills of Ferrous Iodide should be devoid of the smell of iodine.

If a few of the pills be triturated with water, and the liquid be filtered, the filtrate should not assume more than a light blue tint upon the addition of starch T.S. (absence of more than traces of *free iodine*).

Remarks.—These pills are made by triturating reduced iron and iodine until the red tint has disappeared; mixing with glycyrrhiza, sugar, extract of glycyrrhiza, beating the mass to a pilular consistence, and coating the pills with an ethereal solution of balsam of tolu. As already mentioned, these pills and the pills of phosphorus are the only two directed by the pharmacopœia to be coated, and in both cases the coating is not intended to enhance the beauty of the pills, but to prevent oxidation of the active ingredient.

Each pill contains about one grain of ferrous iodide.

Dose.—2 pills.

Ferri Lactas (U. S. P. 1890), $\text{Fe}(\text{C}_3\text{H}_5\text{O}_3)_2$.—This represents the salt of lactic acid, the composition of which will be found on p. 677, made by treating lactic acid with reduced iron, and subsequently evaporating the solution to dryness.

This ferrous preparation is the iron constituent of the formerly official *syrup of hypophosphites with iron*, where it is directed because it is supposed to be a most easily assimilable salt of iron. This claim has never been thoroughly substantiated, and the preparation is one of doubtful value.

It is administered in doses of from 0.6 to 1 Gm.

Ferrous sulphate (FeSO_4), called “green vitriol,” from the Latin *vitreus*, glass, because the crystals look like green glass. When we distil these crystals, an oily liquid passes over, and this, the alchemists of three hundred years since, called “oil of vitriol,” a name that still clings to the distillate—sulphuric acid.

In an impure form ferrous sulphate is called “copperas,” although containing no copper whatever.

Ferrous sulphate is official in three forms: The crystalline, containing seven molecules of water of crystallization; the granulated, which contains the same amount of water as do the crystals (turn to Granulation on p. 148), and, lastly, the exsiccated, which represents the crystalline minus the greater quantity of its water, 100 parts of the crystals yielding 64 parts of the exsiccated. The dried is preferable to the crystalline in the manufacture of pills, being of smaller bulk and not causing a soft mass. Note in these two officials—the crystalline and the dried—an interesting demonstration of the influence of water of crystallization on a substance. Dried ferrous sulphate is a grayish-white, opaque powder; while the crystals, which differ from the dried only by the presence of water of crystallization, are green and transparent.

FERRI SULPHAS. Ferrous Sulphate.

It should contain not less than 99.5 per cent. of pure Ferrous Sulphate [$\text{SO}_2.\text{O}_2\text{Fe} + 7\text{H}_2\text{O}$]; the crystals should not be effloresced and should be kept in well-stoppered bottles.

Large, pale bluish-green, monoclinic prisms, without odor, and having a saline, styptic taste; efflorescent in dry air. On exposure to moist air the crystals rapidly oxidize, and become coated with brownish-yellow, basic ferric sulphate.

Soluble in 0.9 part of water at 25°C . (77°F .) and in 0.3 part of boiling water; insoluble in alcohol.

When slowly heated to 115°C . (239°F .), the crystals disintegrate, and lose 38.87 per cent. of their weight (6 molecules of water of crystallization).

If 1 Gm. of salt be dissolved in about 25 Cc. of water containing 1 Cc. of diluted sulphuric acid, the solution heated to boiling, oxidized with nitric acid, and then mixed with a slight excess of ammonia water, the filtrate from the reddish-brown precipitate produced should be colorless, and on evaporation to dryness and ignition, should not leave any weighable residue (absence of salts of the *alkali metals*).

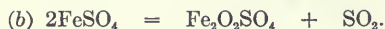
If 1 Gm. of Ferrous Sulphate, in small fragments, be agitated during four or five minutes with 10 Cc. of alcohol, the filtrate should not redden moistened blue litmus-paper (absence of *free acid*).

Tests for Identity.—See Iron and Sulphates in Part V.

Impurities.—Heavy metals. Details in Part V. Salts of the alkaline metals, free acid, see above.

Assay.—Details in Part V.

Remarks.—This salt is made by treating scrap iron with sulphuric acid, and evaporating the solution to a point where the green crystals will be obtained. These green crystals are not very stable, comparatively readily combining with oxygen of the air, with the formation of basic ferric sulphate by the following reactions:



In order to insure the formation of a perfectly green ferrous sulphate, it is usually advisable to crystallize from acid solution containing some card-teeth, the latter acting on the acid with the constant evolution of hydrogen. Thus, cheap copperas can be converted into beautiful crystals of ferrous sulphate by dissolving in water, adding a small quantity of sulphuric acid and some card-teeth, and warming gently until a green solution is obtained. This is filtered from the undissolved ferric oxide and allowed to crystallize.

Ferrous sulphate is used as an astringent and chalybeate tonic.

Dose.—200 milligrammes (3 grains).

FERRI SULPHAS GRANULATUS. Granulated Ferrous Sulphate.

Official recipe, slightly modified, and details of manufacture will be found in Part VII.

Remarks.—This official represents ferrous sulphate in the form of small crystals called granules. It is prepared by dissolving ferrous sulphate in water containing diluted sulphuric acid, evaporating to a certain degree of concentration, and stirring the mixture while it is cooling. (See p. 148.) In this way the salt separates into small crystals, which are immediately transferred to a funnel, stoppered with a plug of cotton, the mother liquid allowed rapidly to drain off,

and the crystals then freed from the remaining adhering water by pouring through a small quantity of alcohol; the object of washing with alcohol being to dry the granules as rapidly as possible, and that without a loss of water of crystallization. It should be emphasized that, theoretically, granulated ferrous sulphate contains exactly the same amount of water as does the crystalline, although in practice it usually loses a small amount of its water of hydration by efflorescence. (2/1)

Dose.—200 milligrammes (3 grains).

FERRI SULPHAS EXSICCATUS. Exsiccated Ferrous Sulphate.

Official recipe and details of manufacture will be found in Part VII.

Remarks.—This preparation is made by efflorescing crystallized ferrous sulphate and then exsiccating on a sand-bath; 100 Gm. of the crystallized yielding 64 to 65 Gm. exsiccated salt.

It will be seen that the exsiccated salt is a much more concentrated preparation than the crystalline, and, therefore, for a similar dose, about three-fifths as much need be given. It is admirably adapted for pills, since the same amount of ferrous sulphate can be given in the exsiccated form in a much smaller bulk. For this reason it is a constituent of the official pills of aloe and iron.

Dose.—125 milligrammes (2 grains).

Ferrous oxalate (U. S. P. 1880), FeC_2O_4 , is made by treating ferrous sulphate with oxalic acid, and this process explains the value of oxalic acid as a remover of ink-stains. Most black ink is tannate of iron, and if to this oxalic acid be added, the tannic acid is replaced by oxalic acid, and the resulting ferrous oxalate leaves a light yellow stain, instead of the pronounced black of the ink, and while the stain is as insoluble as is ink, it does not show. In using oxalic acid for this purpose great care must be taken lest it injure the fabric.

FERRIC SALTS

Having already given the structure of ferric salts our careful consideration, we can proceed to the discussion of the compounds themselves, commencing with one that will thoroughly explain the transition from the ferrous to the ferric state, namely:

FERRI CHLORIDUM. Ferric Chloride.

Ferric Chloride should contain not less than 22 per cent. of metallic iron in the form of chloride.

Recipe.—Solution of Ferric Chloride, one hundred grammes. $\frac{100 \text{ Gm.}}{\text{To make forty grammes.} \quad 40 \text{ Gm.}}$

Evaporate the Solution of Ferric Chloride on a water-bath until it weighs *forty grammes*; then set it aside in a glass-covered vessel, until it forms a crystalline mass. Lastly, break the salt into pieces, and keep it in glass-stoppered bottles, protected from light.

Orange-yellow, crystalline pieces, odorless, or having a faint odor of hydrochloric acid, and a strongly styptic taste.

Very deliquescent in moist air.

Freely and completely soluble in water and in alcohol; also in a mixture of 1 part of ether and 3 parts of alcohol.

At $35.5^\circ \text{C. (96}^\circ \text{F.)}$ the salt fuses to a reddish-brown liquid. When strongly heated it decomposes, losing water and hydrochloric acid, while the anhydrous salt sublimes, leaving a residue of ferric oxide.

Tests for Identity.—See Iron and Chlorides in Part V.

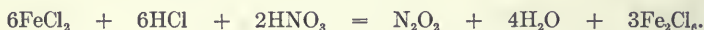
Impurities.—Heavy metals, excess of nitric acid, and ferrous salt. Details in Part V.

Assay.—Details in Part V.

Remarks.—In making this salt, we first treat iron with hydrochloric acid, which gives us ferrous chloride, FeCl_2 , by the following reaction:



It is important to note that usually a metal treated with an acid will yield an “ous” salt of that metal; that salt exhibiting the metal in its lowest valence. Hence we get in this case ferrous chloride, which is analogous to ferrous oxide, which we do not want. We wish ferric chloride, and how are we to get it? How did we make sulphuric oxide from sulphurous oxide? See p. 420: by oxidizing with nitric acid; and that is exactly what we do with ferrous chloride to change it into ferric chloride; the reaction being:



Compare this reaction with that of the conversion of sulphurous oxide to sulphuric oxide, given on p. 420, and the analogy will be noted.

The three molecules of HNO_3 break into N_2O_2 , H_2O , and three atoms of oxygen, and on the basis of the three atoms of oxygen, yielded by the two molecules of nitric acid, hangs the entire proportion of the equation.

Note that in this oxidation the oxygen does not combine with the iron, but that it seizes the hydrogen of the hydrochloric acid, setting the chlorine free and giving it a chance to combine with the iron of the ferrous chloride. This is a confirmation of the assertion on p. 420 that the function of an oxidizing agent is not merely that of adding oxygen to a substance, but that its main effort is toward raising the valence of one of the elements entering into the reaction. Moreover, the element whose valence is raised is not necessarily the one which is oxidized.

The pharmacopœial statement that ferric chloride is soluble in a mixture of ether and alcohol is made use of in Rothe's process for the quantitative estimation of iron.

The official ferric chloride is used as a styptic externally, and internally as a tonic.

Dose.—65 milligrammes (1 grain).

LIQUOR FERRI CHLORIDI. Solution of Ferric Chloride.

An aqueous solution of Ferric Chloride, which should contain not less than 29 per cent. of the anhydrous salt [$\text{FeCl}_3 = 161.04$], corresponding to 10 per cent. of metallic iron.

Recipe.—Iron, in the form of fine, bright wire, and cut into small pieces, one hundred and twenty-five grammes. 125 Gm.
 Hydrochloric Acid, six hundred and eighty grammes 680 Gm.
 Nitric Acid,
 Distilled Water, each, a sufficient quantity,
 To make one thousand grammes. . 1000 Gm.

Introduce the Iron Wire into a flask having a capacity of about *two thousand cubic centimeters*, pour upon it a mixture of *four hundred and twenty grammes* of Hydrochloric Acid and *two hundred and fifty cubic centimeters* of Distilled Water, and heat upon a water-bath for not less than one hour and fifteen minutes, or until effervescence ceases; then boil the liquid, filter it through paper, and, having rinsed the flask and Iron Wire with a little hot Distilled Water, pass the rinsings through the filter. To the filtered liquid add *two hundred and twenty grammes* of Hydrochloric Acid, add the mixture slowly and gradually, in a stream, to *sixty-five grammes* of Nitric Acid contained in a capacious porcelain vessel, and warm gently. After effervescence ceases, apply heat, by means of a sand-bath, stirring occasionally, until the liquid is free from Nitric Acid. If the solution has acquired a black color, continue the addition of Nitric Acid, drop by drop, until red fumes are no longer evolved and the solution assumes a clear reddish-brown color. Finally, add the remaining *forty grammes* of Hydrochloric Acid and enough Distilled Water to make the Solution weigh *one thousand grammes*.

A reddish-brown liquid, having a faint odor of hydrochloric acid, an acid, strongly styptic taste, and an acid reaction.

Specific gravity: about 1.315 at 25° C. (77° F.).

If the iron be completely precipitated from a portion of the Solution by an excess of ammonia water, the filtrate should be colorless, and should not yield a precipitate with hydrogen sulphide T.S. (absence of *zinc* or *copper*); nor should it leave a fixed residue on evaporation and gentle ignition (absence of *salts of the fixed alkalies*).

If to 3 drops of Solution of Ferric Chloride, 10 Cc. of tenth-normal sodium thio-sulphate V.S. be added, and then slowly heated to boiling, no brownish-red precipitate of ferric hydroxide should separate (absence of *oxychloride*).

Tests for Identity.—See Iron and Chlorides in Part V.

Impurities.—Zinc, copper, fixed alkaline salts, and oxychloride, see above. Nitric acid and ferrous salts. Details in Part V.

Assay.—Details in Part V.

This preparation contains 29 per cent. of anhydrous salt, the solution containing an excess of free hydrochloric acid. It is rarely administered in medicine, the tincture of ferric chloride being preferred for internal use, and Monsel's solution is better for external styptic purposes.

Dose.—0.1 Cc. (1½ minims).

TINCTURA FERRI CHLORIDI. Tincture of Ferric Chloride.

A hydro-alcoholic solution of Ferric Chloride [$\text{FeCl}_3 = 161.04$], containing not less than 13.28 per cent. of the anhydrous salt, corresponding to 4.6 (4.58) per cent. of metallic iron.

Recipe.—Solution of Ferric Chloride, *three hundred and fifty cubic centimeters*. 350 Cc.
Alcohol, *a sufficient quantity*,
To make *one thousand cubic centimeters*. . . 1000 Cc.

Mix the Solution with enough Alcohol to make *one thousand cubic centimeters*. Let the Tincture stand, in a closely covered vessel, protected from light, at least three months; then transfer it to glass-stoppered bottles, and keep it protected from light.

A bright, brownish liquid, having a slightly ethereal odor, a very astringent, styptic taste, and an acid reaction.

Specific gravity: about 1.005 at 25° C. (77° F.).

After the Tincture has been exposed for some time to daylight, it yields a greenish or greenish-blue color with potassium ferricyanide T.S., showing the presence of some ferrous salt, due to reduction.

Tests for Identity.—See Iron and Chlorides in Part V.

Impurity.—Nitric acid. Details in Part V.

Assay.—Details in Part V.

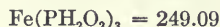
Remarks.—This galenic preparation is made by combining 350 Cc. of solution of ferric chloride with alcohol, to make 1000 Cc., and allowing same to stand for three months before dispensing, in order that the free hydrochloric acid found in the solution will have time to act on the alcohol and form compound ethers, which render it more active.

The tincture of ferric chloride is one of the most satisfactory means for the administration of iron. The chief objection is the fact that, when taken directly into the mouth, it is apt to injure the teeth, due partly to the iron, but chiefly to the excess of acid present in the preparation. It is, therefore, generally directed to be administered largely diluted with water and sucked through a tube. A very simple and effective method of administration, totally devoid of any danger of injuring the teeth, is to drop the dose of the tincture into a gelatin capsule of appropriate size, say, No. 2 or No. 3, and then swallowing same immediately in a draught of water. Such capsules are unstable, the water in the tincture dissolving the gelatin, and hence must be made by the patient at the moment of administration—a very simple matter when the proper directions are given.

Dose.—0.5 Cc. (8 minims).

The tincture of ferric chloride is an ingredient of the *solution of iron and ammonium acetate*. (See p. 565.)

FERRI HYPOPHOSPHIS. Ferric Hypophosphite.



It should contain not less than 98 per cent. of pure Ferric Hypophosphite [$(\text{PH}_2\text{O}_2)_3\text{Fe}$] and should be kept in well-stoppered bottles.

A white or grayish-white powder, odorless and nearly tasteless; permanent in the air.

Soluble in 2300 parts of water at 25° C. (77° F.) and in 1200 parts of boiling water; more readily soluble in the presence of hypophosphorous acid, or in a warm, concentrated solution of an alkali citrate, forming with the latter a green solution.

When strongly heated in a test-tube the salt evolves spontaneously inflammable hydrogen phosphide gas, and, on complete ignition, leaves a residue of ferric pyrophosphate.

Ferric Hypophosphite is readily oxidized by nitric acid or other oxidizing agents.

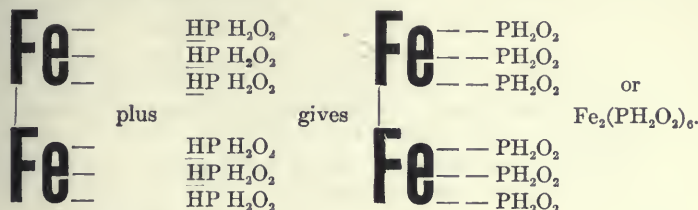
Tests for Identity.—See Iron and Hypophosphites in Part V.

Impurities.—Carbonate, calcium, phosphate, and heavy metals. Details in Part V.

Ferric hypophosphite, $\text{Fe}(\text{PH}_2\text{O}_2)_3$, seems to present a very difficult symbolic formula. However, a complete explanation of the formula of hypophosphorous acid, HPH_2O_2 , is given on p. 430. The formula there given shows but one hydroxyl group, and a dictum in chemistry is that in all inorganic acids, save the haloids, only those hydrogen atoms found in a hydroxyl group are replaceable by a metal. Hence, hypophosphorous acid has but one replaceable hydrogen; unlike its near relative, phosphoric acid, whose graphic formula, given on p. 373, shows it to have three hydroxyls, and therefore three replaceable hydrogens.

Since one molecule of hypophosphorous acid contains but one atom of replaceable hydrogen, and since the valence of ferric iron is III, or, if preferred, $(\text{Fe}_2)^{\text{VI}}$, two atoms of ferric iron, which can

replace six atoms of hydrogen, will combine with six molecules of hypophosphorous acid, thus:



This official salt is made by treating a solution of calcium hypophosphite with a solution of ferric chloride, when ferric hypophosphite is produced by the following reaction:



In this case calcium chloride dissolves, while the ferric hypophosphite precipitates.

Ferric hypophosphite possesses properties similar to all hypophosphites, with the addition of the advantage of being a chalybeate tonic.

Dose.—200 milligrammes (3 grains).

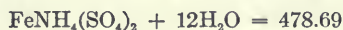
Ferric valerianate (U. S. P. 1890), $\text{Fe}(\text{C}_5\text{H}_9\text{O}_2)_3$, is the ferric salt of valerianic acid, the chemistry of which will be found on p. 688. It is made by the double decomposition of sodium valerianate with ferric sulphate, the reaction being as follows:



The insoluble ferric valerianate thus produced is separated from the sodium sulphate by filtration and subsequent washing of the precipitate.

The ferric valerianate is given as a tonic and nervine in doses of from 0.3–0.6 Gm. (5–10 grains).

FERRI ET AMMONII SULPHAS. Ferric Ammonium Sulphate.



It should contain, in the uneffloresced condition, 99.5 per cent. of pure Ferric Ammonium Sulphate and not less than 11.5 per cent. of metallic iron. It should be kept in well-stoppered bottles, protected from light.

Pale violet, octahedral crystals, without odor, and having an acid, styptic taste; efflorescent on exposure to the air.

Soluble in 2.7 parts of water at 25° C. (77° F.) and in 0.8 part of boiling water; insoluble in alcohol.

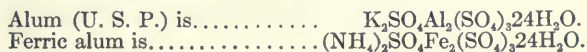
When strongly heated the crystals fuse, lose their water of crystallization, expand, and finally leave a pale brown residue.

Tests for Identity.—See Iron, Ammonium, and Sulphates in Part V.

Impurities.—Aluminum and chlorides. Details in Part V.

Assay.—Details in Part V.

Remarks.—*Ferric ammonium sulphate* is the well-known *ferric alum*, the chemistry of which was thoroughly explained on p. 536, among the other alums, hence we only need to emphasize the analogy between the official alum and ferric alum:



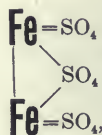
As already explained under Alum (p. 535), the present pharmacopœia gives both alums, the above formulæ divided by two.

Ferric alum is made by combining molecular quantities of the solution of ferric sulphate with a solution of ammonium sulphate. On evaporating the mixture the pure crystals of ferric alum separate, leaving the dark-brown mother liquor. As already mentioned on p. 536, ferric alum possesses very few of the properties of the average iron salt, being much more similar to the alums.

It is used as styptic and very rarely internally.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Ferric sulphate, $\text{Fe}_2(\text{SO}_4)_3$, which is graphically



is official in the form of a *solution* containing 36 per cent. of ferric sulphate—called liquor ferri tersulphatis, to distinguish it from the official solution of the basic salt, *liquor ferri subsulphatis*, which is commonly called “Monsel’s solution.”

LIQUOR FERRI TERSULPHATIS. Solution of Ferric Sulphate.

An aqueous solution, which should contain about 36 per cent. of normal Ferric Sulphate [$\text{Fe}_2(\text{SO}_4)_3=397.05$], corresponding to not less than 10 per cent. of metallic iron.

A dark reddish-brown liquid, almost odorless, having an acid, strongly styptic taste, and an acid reaction.

Specific gravity: about 1.432 at 25°C . (77°F .).

Miscible with water and alcohol, in all proportions, without decomposition.

On slowly mixing 2 volumes of the Solution with 1 volume of concentrated sulphuric acid, in a beaker, no solid white mass should separate on standing (difference from *subsulphate*).

Recipe and details of manufacture in Part VII.

Tests for Identity.—See Iron and Sulphates in Part V.

Impurities.—*Subsulphate*, see above. Nitric acid and ferrous salt. Details in Part V.

Assay.—Details in Part V.

Remarks.—Solution of ferric sulphate is made by the oxidation of ferrous sulphate with nitric acid, and by a process entirely analogous to that described under the head of ferric chloride. Details on p. 556. As in the case of ferric chloride, it is seen that, to accomplish this result, not only is nitric acid necessary, but also an additional quantity of the characteristic acid, in this case sulphuric acid, the amount of the latter being directly proportional to the difference in SO_4 groups expressed in the two formulas, ferrous sulphate and ferric sulphate.

The solution of tersulphate of iron is one of the most important of the iron preparations, although very rarely called for in prescriptions or at the counter. It is the basis of all the scale salts of iron, and is the iron constituent used in preparing the antidote for arsenic. For this reason, if for no other, the pharmacist must keep in stock a solution of tersulphate of iron.

It is worth noting that the ferric sulphate of this preparation is the true persulphate of iron, though, through an unfortunate blunder, custom has established the precedent of calling the solution of subsulphate of iron "solution of persulphate of iron."

LIQUOR FERRI SUBSULPHATIS. Solution of Ferric Subsulphate.

An aqueous solution of variable chemical composition, containing an amount of basic ferric sulphate corresponding to not less than 13.57 per cent. of metallic iron.

<i>Recipe.</i> — Ferrous Sulphate , in clear crystals, <i>six hundred and seventy-five grammes</i>	675 Gm.
Sulphuric Acid , <i>sixty-five grammes</i>	65 Gm.
Nitric Acid ,	
Distilled Water , each, <i>a sufficient quantity</i> ,	
To make <i>one thousand grammes</i> ..	1000 Gm.

Add the Sulphuric Acid to *five hundred cubic centimeters* of Distilled Water in a capacious porcelain dish, heat the mixture to nearly 100° C. (212° F.), then add *seventy grammes* of Nitric Acid, and mix well. Divide the Ferrous Sulphate, coarsely powdered, into four equal portions, and add these portions, one at a time, to the hot liquid, stirring after each addition until effervescence ceases. If, after the Ferrous Sulphate has been dissolved, the solution is of a black color, add Nitric Acid, a few drops at a time, with heating and stirring, until it no longer causes red fumes to be evolved; then boil the Solution until it assumes a ruby-red color and is free from nitric acid. Lastly, add enough Distilled Water to make the product weigh *one thousand grammes*. Filter if necessary. Keep it in well-stoppered bottles, in a moderately warm place (not under 22° C. or 71.6° F.), protected from light.

This solution sometimes crystallizes, forming a semisolid, whitish mass. When this occurs, the application of a gentle heat to the bottle will restore the liquid condition.

A dark reddish-brown liquid, odorless or nearly so, of an acid, strongly styptic taste, and an acid reaction.

Specific gravity: about 1.548 at 25° C. (77° F.).

Miscible with water and alcohol, in all proportions, without decomposition.

On slowly mixing 2 volumes of the Solution with 1 volume of concentrated sulphuric acid, in a beaker, a semisolid white mass should separate on standing (difference from *tersulphate*).

Tests for Identity.—See Iron and Sulphates in Part V.

Impurities.—Tersulphate, see above. Nitric acid and ferrous salt. Details in Part V.

Assay.—Details in Part V.

Remarks.—This is commonly called Monsel's solution. The prefix "sub" means a basic salt—a combination of an oxide of a metal with a salt of the same metal. Thus, ferric *subsulphate* is a combination of ferric sulphate and ferric oxide; bismuth *subnitrate* is a combination of bismuth nitrate and bismuth oxide; and so on. Note that in the pharmacopœia of 1900, no symbolic formula is assigned Monsel's solution, it being described as of "variable chemical composition." In the pharmacopœia of 1880 the formula was given as $\text{Fe}_4\text{O}(\text{SO}_4)_5$, which may be untangled as follows:

$$\begin{array}{rcl}
 \text{If we take three molecules } \text{Fe}_4\text{O}(\text{SO}_4)_5 \text{ we get.....} & \text{Fe}_{12}\text{O}_3(\text{SO}_4)_{15} \\
 \text{Subtract from this.....} & \text{Fe}_2\text{O}_3 \\
 \text{We have left.....} & \text{Fe}_{10}(\text{SO}_4)_{15} \\
 & \text{Or.....} 5\text{Fe}_2(\text{SO}_4)_3.
 \end{array}$$

In other words, five molecules of ferric sulphate and one molecule of ferric oxide give three molecules of ferric subsulphate.

Both solutions—of ferric subsulphate and of ferric tersulphate—are made from ferrous sulphate, just as ferric chloride was made from ferrous chloride—by oxidation with nitric acid, the reaction in making ferric tersulphate being:



Compare this to the equation in making subsulphate, viz.:



This shows that while three molecules of sulphuric acid are used in both reactions, that quantity suffices for six molecules of ferrous sulphate in making the tersulphate, and for twelve molecules of ferrous sulphate when the subsulphate is made. In other words, in making the tersulphate from a given quantity of ferrous sulphate, twice as much sulphuric acid is required as is needed to make the subsulphate. The N_2O_2 of the equations just given is a colorless gas, which on coming in contact with the air is instantly converted into N_2O_4 , which we met in the sulphuric acid manufacture and which form the red fumes spoken of in the pharmacopœial process.

Note that Monsel's solution—the subsulphate—is to be dispensed when the persulphate is called for.

As just mentioned above, the solution of subsulphate of iron differs from the solution of tersulphate of iron in that it is made from less sulphuric acid and more ferrous sulphate. This is best shown by a comparison of the quantities of the ingredients of the two solutions:

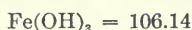
	FERROUS SULPHATE.	SULPHURIC ACID.
Solution of tersulphate.....	500 Gm.	96 Gm. }
Solution of subsulphate.....	675 Gm.	65 Gm. } to 1000 Gm. finished product.

In comparing the pharmacopœial description of the two solutions, it will be seen that the simplest test of distinction is the behavior of each when treated with an equal quantity of concentrated sulphuric acid. Under such treatment the subsulphate forms a semisolid mass, while the tersulphate forms a clear, limpid liquid.

The solution of subsulphate of iron is a favorite styptic and astringent, used for staunching the flow of blood, and is rarely given internally. The residue from the evaporation of the solution, termed *Monsel's powder* in commercial usage, is very largely used for this purpose, especially in barber shops for the drying up of cuts produced in shaving.

Dose.—0.2 Cc. (3 minims).

FERRI HYDROXIDUM. Ferric Hydroxide.



[FERRI OXIDUM HYDRATUM, PHARM. 1890]

Recipe.—Solution of Ferric Sulphate, one hundred cubic centimeters..... 100 Cc.
 Ammonia Water, one hundred and thirty-eight cubic centimeters..... 138 Cc.
 Water, a sufficient quantity.

To the Ammonia Water, previously diluted with *five hundred cubic centimeters* of cold water, add, with constant stirring, the Solution of Ferric Sulphate, previously diluted with *one thousand cubic centimeters* of cold Water. As soon as the precipitate has subsided, draw off the clear liquid by means of a siphon, then mix the precipitate intimately with about *one thousand cubic centimeters* of cold Water, again draw off the clear liquid after subsidence of the precipitate, and repeat this operation, until a portion of the decanted liquid gives not more than a slight cloudiness with barium chloride test solution. Finally, transfer the precipitate to a wet muslin strainer, and, after it has drained, mix it with sufficient cold Water to make the mixture weigh *three hundred grammes*.

Ferric Hydroxide, thus prepared, is a brownish-red magma, wholly soluble in hydrochloric acid without effervescence.

Remarks.—Ferric hydroxide, or ferric hydrate ($\text{Fe}(\text{OH})_3$), is formed by the following reaction:



It is a red-brown, thick, tenacious precipitate, called a "magma," already mentioned (p. 141).

In making this official, it is very important that the tersulphate solution be added to the diluted ammonia. If the reverse operation is performed, we obtain an oxyhydrate rather than the hydroxide itself.

Since ferric hydroxide very rapidly decomposes into water, and ferric oxide, which is insoluble even in acids, it must be freshly prepared when wanted, and since when needed for poisoning with arsenic, it must be dispensed with the least possible loss of time, every pharmacist should keep on hand diluted solutions of ammonia water and of ferric sulphate. Moreover, in order to expedite matters, it is advisable to place convenient to the bottles holding these two solutions strainers and their required paraphernalia. Since the dispensing of these preparations for poisoning by arsenic, fortunately, does not occur every day, this outfit may rest on the shelf for years without being used. Such being the case, it is advisable to renew, from time to time, the diluted ammonia water. The diluted solution of ferric sulphate keeps perfectly. When dispensed as an antidote for arsenic poisoning, haste is the prime requirement, and hence the pharmacist need not bother about washing the precipitate completely free from the ammonium sulphate. All that is necessary is to free the magma from ammonia water. In practice it is best to prepare rapidly a small quantity of the magma, say, one or two ounces, in a very incomplete way, and despatch this to the sufferer with the promise of an additional quantity to be sent later on. This additional quantity can be prepared with more regard to pharmacopœial requirements. However, for the purpose of an antidote for arsenic the ferric hydroxide with magnesia is preferred.

For chemical purposes, such as for the manufacture of scale salts and the detannating of pharmaceuticals, the ferric hydroxide so employed should be totally free from ammonium sulphate. The process of washing the precipitate is very tedious. The writer, for such purposes, throws the magma on large canvas strainers of texture sufficiently coarse to permit the passage of water. Through such strainers, however, a considerable quantity of the magma is apt to escape at first, and in order that none of this be lost, the strainers are arranged

in a rack, so that tiers of at least three such strainers are employed. In this way the magma passing through the top strainer had a chance to collect on the second strainer, and that which passed through the second strainer was almost sure to be retained by the third one. Beneath the third strainer a suitable receptacle for collecting the drippings was placed. After the precipitate, with its supernatant liquor, had been transferred to the strainers, the wash-water was introduced into the top strainer by means of a siphon, so regulated that the amount of water dripping through exactly replaces the amount of water dripping therefrom.

In this way large quantities of magma can be readily washed within twelve to eighteen hours.

Ferric hydroxide was introduced into medicine by reason of its value as an antidote for arsenic, the discovery of this property being made by Bunsen and Berthold in 1834. The chemical reason of the efficiency of the hydroxide for this purpose is very simple, being due to the formation of the insoluble ferric arsenite, as shown in the following equation:



For antidotal purposes it is administered in tablespoonful doses, followed by an emetic, and, as mentioned above, the ferric hydroxide with magnesia is now considered preferable.

Pharmaceutically, ferric hydroxide is the basis of the scale preparations of iron. It is also the iron constituent of the lozenges of iron and the plaster of iron.

Dialyzed iron is an unofficial modification of ferric hydroxide, and has already been discussed as a type of the products of dialysis (p. 150).

This preparation was originally made by treating solution of ferric chloride with freshly precipitated ferric hydroxide. It is now usually made by treating solution of ferric chloride with ammonia water. The mixture is placed on the dialyzing membrane, when crystalline substances, ammonium chloride and ferric chloride, pass through the septum, leaving colloidal ferric oxychloride, of composition varying from Fe_2Cl_6 , $11\text{Fe}_2\text{O}_3$ to Fe_2Cl_6 , $31\text{Fe}_2\text{O}_3$.

Dialyzed Iron is a limpid, red-brown liquid, free from acidity and without styptic taste. It is used as a tonic in doses of 5 to 30 drops.

FERRI HYDROXIDUM CUM MAGNESII OXIDO. Ferric Hydroxide with Magnesium Oxide.

[FERRI OXIDUM HYDRATUM CUM MAGNESIA, PHARM. 1898]

Recipe.—Solution of Ferric Sulphate, forty cubic centimeters. 40 Cc.
Magnesium Oxide, ten grammes. 10 Gm.
Water, a sufficient quantity.

Mix the Solution of Ferric Sulphate with *one hundred and twenty-five cubic centimeters* of Water, and keep the liquid in a large, well-stoppered bottle. Rub the Magnesium Oxide with cold Water to a smooth and thin mixture, transfer this to a bottle capable of holding about *one thousand cubic centimeters*, and fill it with Water to about three-fourths of its capacity. When the preparation is wanted for use, shake the Magnesium Oxide mixture to a homogeneous, thin magma, add it gradually to the diluted Solution of Ferric Sulphate, and shake them together until a uniform, smooth mixture results.

NOTE.—For the rapid preparation of this antidote to arsenical poisoning the diluted Solution of Ferric Sulphate, and the mixture of Magnesium Oxide with Water, should always be kept on hand, in separate bottles, ready for immediate use.

It will be seen that this preparation is made by treating diluted solution of ferric sulphate with the milk of magnesia (that is, magnesia rubbed with water), the reaction being:



The result of this reaction, as will be noticed, is practically the same as in the official method of making pure ferric hydroxide. The great advantage, however, is that the magma thus produced is efficient without washing, because the impurities are the mild alkali, magnesium hydroxide, and the harmless magnesium sulphate. These two compounds also serve antidotal purposes by reason of the formation of the insoluble magnesium arsenite. For this reason this preparation is greatly to be preferred over the pure ferric hydroxide as an antidote for arsenic, it being rapidly prepared by the blending of the diluted solution of ferric sulphate with a magnesia mixture, followed by the rapid removal of the superfluous water by straining. Note that the diluted solutions of ferric sulphate and the milk of magnesia should be kept on hand by the pharmacist, for instant admixture in cases of arsenical poisoning.

Dose.—Arsenical antidote, 120 Cc. (4 fluidounces).

LIQUOR FERRI ET AMMONII ACETATIS. Solution of Iron and Ammonium Acetate.

<i>Recipe.</i> —Tincture of Ferric Chloride, forty cubic centimeters.	40 Cc.
Diluted Acetic Acid, sixty cubic centimeters.....	60 Cc.
Solution of Ammonium Acetate, five hundred cubic centimeters.....	500 Cc.
Aromatic Elixir, one hundred and twenty cubic centimeters.....	120 Cc.
Glycerin, one hundred and twenty cubic centimeters.	120 Cc.
Water, a sufficient quantity,	
To make one thousand cubic centimeters..	1000 Cc.

To the Solution of Ammonium Acetate (which should not be alkaline) add, successively, the Diluted Acetic Acid, the Tincture of Ferric Chloride, the Aromatic Elixir, and the Glycerin, and, lastly, enough Water to make the product measure one thousand cubic centimeters.

This preparation should be freshly made when wanted.

Remarks.—In mixing the chemicals as above described, ferric acetate is produced, and a brilliant garnet solution ensues. In preparing the solution, it is important that the full amount of acetic acid be employed, for if the solution is not distinctly acid, the basic acetate of iron will separate as an unsightly red-brown precipitate.

The same precipitate occurs in a properly prepared solution, when it becomes old, hence the pharmacopœial direction as to preparing freshly.

Basham's mixture, as this solution is called, is a popular tonic in some sections.

Dose.—16 Cc. (4 fluidrachms).

Solution of Ferric Acetate (U. S. P. 1890).—This preparation, containing 31 per cent. of the anhydrous salt, is made by dissolving freshly prepared hydroxide in a molecular quantity of glacial acetic acid.

The solution of ferric acetate is used as a mild chalybeate tonic, and in doses of 2 to 10 minims.

Solution of Ferric Nitrate (U. S. P. 1890).—This former official, containing 6.2 per cent. of anhydrous salt, is made by a process very similar to that used in making the solution of ferric acetate; the ferric hydroxide in this case being dissolved in a molecular quantity of nitric acid. This solution is interesting by reason of the light tint which it possesses; all other ferric solutions are of a brilliant garnet-red, but this one usually possesses a color scarcely darker than amber.

Solution of Ferric Citrate (U. S. P. 1890).—Recipe and details of manufacture in Part VII.

This solution, like the two just discussed, is made by treating ferric hydroxide with an appropriate acid, in this case, citric acid. If to the drained magma crystals of citric acid are added, and the mixture placed on a water-bath for a few minutes, the magma is converted into a clear garnet-red solution, usually weaker than the pharmacopœial strength. It is, therefore, evaporated on a water-bath at a temperature not exceeding 60° C. This shows that a large amount of water is mechanically held by the ferric hydroxide, its absorbing capacity being almost as great as that of aluminum hydroxide.

The solution of ferric citrate was recognized by the pharmacopœia of 1890, because affording a convenient liquid form of that valuable remedy—ferric citrate. It contains about 50 per cent. of ferric citrate, and will be found of great convenience in dispensing in prescriptions directing ferric citrate dissolved in water, since the dissolving of ferric citrate is quite tedious. It seems unfortunate that in the eighth revision of the pharmacopœia this solution was dropped.

THE SCALE SALTS OF IRON

Having discussed the official ferrous and ferric salts, we can now consider the third group of official iron compounds—the scale preparations of iron.

These preparations are not always definite chemicals; thus, the official soluble ferric pyrophosphate is a mixture of pyrophosphate of iron, which in pure form is an insoluble green-gray powder, and sodium citrate, with traces of sodium pyrophosphate and ferric citrate. The scale preparations are bright, shining fragments, which are not crystals, despite an impression to that effect, arising no doubt from a brand of scale pepsin on the market termed “crystal pepsin.”

Crystals are bodies having definite geometric form, and any examination of a scale preparation, no matter how superficial, will prove absence of distinct unvarying form. Preparations are scaled just as molasses candy is made—by concentrating to a thick syrup and pouring this on a smooth hard surface, usually on sheets of glass.

All scale preparations of iron are made directly or indirectly from ferric hydroxide, that red-brown magma formed by adding solution of ferric sulphate to a diluted water of ammonia.

If we treat the magma with citric acid, ferric citrate is formed; if tartaric acid be the acid added, we get ferric tartrate; while if we use acid potassium tartrate (potassium bitartrate), iron and potassium tartrate will be the product.

All the official scale preparations of iron save the last two mentioned are made from ferric citrate; hence we will discuss those two first, and then have clear sailing for those owing their existence to ferric citrate.

FERRI ET AMMONII TARTRAS. Iron and Ammonium Tartrate.

It should contain Iron and Ammonium Tartrate corresponding in amount to not less than 13 per cent. of metallic iron, and should be kept in well-stoppered bottles, protected from light.

Thin, transparent scales, varying in color from garnet-red to reddish-brown, without odor, and having a sweetish, slightly ferruginous taste; slightly deliquescent in the air. Very soluble in water; insoluble in alcohol.

When strongly heated the salt chars, emits fumes having the odor of burning sugar, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction upon red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

An aqueous solution of Iron and Ammonium Tartrate should be neutral to litmus-paper, and is not precipitated, but rendered darker in color, by ammonia water.

Tests for Identity.—See Iron, Ammonium, and Tartrates in Part V.
Impurities.—Alkaline citrates and tartrates. See above.

Assay.—Details in Part V.

Remarks.—As ferric tartrate itself is very sparingly soluble in water, it is replaced in the pharmacopœia by the more soluble *iron and ammonia tartrate*, which is prepared by adding ferric hydroxide to a solution of ammonium tartrate and tartaric acid, evaporating to syrupy consistence, and pouring on plates of glass. As the last part of the process—evaporating and pouring on plates of glass—is common to all scale preparations, we will, for brevity's sake, call it scaling in future. Like all other scaled iron salts, the product is a tonic.

Dose.—250 milligrammes (4 grains).

FERRI ET POTASSII TARTRAS. Iron and Potassium Tartrate.

It should contain Iron and Potassium Tartrate corresponding in amount to not less than 15 per cent. of metallic iron, and should be kept in well-stoppered bottles, protected from light.

Thin, transparent scales, varying in color from garnet-red to reddish-brown, without odor, and having a sweetish, ferruginous taste; slightly deliquescent in the air.

Very soluble in water; insoluble in alcohol.

When strongly heated the salt chars, emits fumes having an odor resembling that of burning sugar, and finally leaves a dark brown residue, having a strongly alkaline reaction, and effervescing with acids (distinction from *iron and ammonium tartrate*).

An aqueous solution of Iron and Potassium Tartrate should be neutral to litmus-paper, and is not precipitated, but rendered darker in color, by ammonia water.

Tests for Identity.—See Iron, Potassium, and Tartrates in Part V.
Impurities.—Iron and ammonium tartrate. See above.

Assay.—Details in Part V.

Remarks.—The manufacture of this chemical is very similar to that of the one just discussed. In the iron and the ammonium tartrate the ferric hydroxide is treated with what may be considered a solution of ammonium bitartrate, whereas in the iron and potassium tartrate the magma is dissolved by treating with potassium bitartrate or cream of tartar. This fact is worth passing notice, inasmuch as it clearly shows that acid salts possess acid properties; that is, that the hydrogen found in acid salt is capable of combining with a metal, just as does the hydrogen with the acid from which it is derived.

Besides the official preparation, a commercial form of iron and potassium tartrate formerly enjoyed large popularity. This was the

chemical in the form of an oval lump, to one end of which was attached a loop of string or ribbon. This form was formerly highly esteemed in France under the name of "*boule de Mars*." This name, by the way, suggested the alchemistic title of the element iron, which was called, by the forerunners of chemistry, after the god of war, Mars. Another survival of the alchemistic name of iron is shown in the words "*crocus Martis*." The "*boule de Mars*" was supposed to be carried around by the patient, and when a chalybeate tonic was desired, the lump was suspended into a glass of water or wine, and after sufficient immersion it was supposed that a sufficient quantity of the chemical would be dissolved in the fluid, thus producing a pleasant tonic.

The official iron and potassium tartrate is used as a tonic.

Dose.—250 milligrammes (4 grains).

FERRI CITRAS. Ferric Citrate.

It should contain Ferric Citrate corresponding in amount to not less than 16 per cent. of metallic iron, and should be kept in well-stoppered bottles, protected from light.

Thin, transparent, garnet-red scales, without odor, and having a slightly ferruginous taste.

Slowly but completely soluble in water at 25° C. (77° F.) and readily soluble in hot water, but diminishing in solubility with age; insoluble in alcohol.

When strongly heated the salt chars, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction upon red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

An aqueous solution of the salt shows an acid reaction with blue litmus-paper, and is not precipitated, but rendered darker in color, by ammonia water.

With potassium ferrocyanide T.S. the aqueous solution yields a bluish-green color or precipitate, which is increased and rendered dark blue by the subsequent addition of hydrochloric acid (difference from *iron and ammonium citrate*).

Tests for Identity.—See Iron and Citric Acid in Part V.

Impurities.—Alkaline citrates and tartrates, iron, and ammonium citrate. See above. Tartrates, details in Part V.

Assay.—Details in Part V.

Recipe and details of manufacture in Part VII.

Remarks.—This preparation is made by evaporating a solution of ferric citrate at a temperature not higher than 60° C. to a thin syrup, and then scaling this by pouring it on a glass plate.

Ferric citrate is very soluble in water; its solution, however, takes place very slowly, as is also true of most of the scale salts of iron.

In dissolving these salts they should never be reduced to powder, nor is it advisable to triturate in a mortar with a solvent. The solution is effected far more rapidly by pouring the scale salts upon the full amount of solvents contained in a bottle and vigorously shaking.

But even thus, the solution of the salt, requires considerable patience, hence the use of a solution of ferric citrate is advisable wherever possible. The very fact that ferric citrate and other scale salts slowly dissolve renders them valuable for the manufacture of pills, because, when blended with an excipient, they are apt to retain their form, producing the pills that do not flatten.

Ferric citrate is a chalybeate tonic.

Dose.—250 milligrammes (4 grains).

FERRI ET AMMONII CITRAS. Iron and Ammonium Citrate.

It should contain Iron and Ammonium Citrate corresponding in amount to not less than 16 per cent. of metallic iron, and should be kept in well-stoppered bottles, protected from light.

Thin, transparent, garnet-red scales, without odor, and having a saline, mildly ferruginous taste; deliquescent in moist air.

Readily and completely soluble in water, but insoluble in alcohol.

When strongly heated the salt chars, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction with red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

An aqueous solution of the salt is neutral to litmus-paper.

The aqueous solution is not precipitated, but rendered darker in color, by ammonia water.

With potassium ferrocyanide T.S. the solution does not afford a blue color or precipitate, unless it be acidulated with hydrochloric acid (difference from *ferric citrate*).

Tests for Identity.—See Iron, Ammonium, and Citrates in Part V.

Impurities.—Ferric citrate. See above. Tartrate. Details in Part V.

Assay.—Details in Part V.

Remarks.—Ferric citrate is very slowly soluble in cold water; hence a more soluble form, made by adding ammonia water to solution of ferric citrate and scaling, is official under the name of *iron and ammonium citrate*. It is a curious fact that while ammonia water precipitates the crystalline ferrous and ferric salts, it simply darkens and renders more soluble the scale preparations.

While ferric citrate occurs in garnet-red scales, the scales of iron and ammonium citrate are yellow-brown. Iron and ammonium citrate is the ferruginous constituent of *wine of ferric citrate*, to which refer.

Medical Properties.—Chalybeate tonic.

Dose.—250 milligrammes (4 grains).

FERRI ET QUININÆ CITRAS. Iron and Quinine Citrate.

It should contain not less than 11.5 per cent. of dried quinine, and ferric citrate corresponding in amount to not less than 13.5 per cent. of metallic iron. It should be kept in well-stoppered bottles, protected from light.

Thin, transparent scales, of a reddish-brown color, without odor, and having a bitter, mildly ferruginous taste; slowly deliquescent.

Slowly, but completely soluble in cold water, more readily soluble in hot water, partially soluble in alcohol. Its solubility is diminished by age.

When strongly heated the salt chars, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction upon red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

The aqueous solution of the salt shows an acid reaction with blue litmus-paper.

Tests for Identity.—See Iron and Citrates in Part V. *Test for quinine* gives curdy white precipitate on adding ammonia water.

Impurities.—Alkaline citrates and tartrates, given above. Tartrate. Details in Part V.

Assay.—Details in Part V.

Remarks.—No process for the manufacture of this salt is given by the present pharmacopœia. The pharmacopœia of 1890 directed its manufacture by adding citric acid and the uncombined alkaloid, quinine, to a solution of ferric citrate, and then scaling the resulting liquid.

The dried scales are of a reddish-brown tint.

This is important because of the fact that a very large amount of the commercial iron and quinine citrate possesses a yellowish-brown tint, and is none other than soluble iron and quinine citrate.

For practical purposes the latter preparation may be preferable to the official iron and quinine citrate, but the point at issue is that if a physician prescribes iron and quinine citrate, the soluble iron and quinine citrate should not be dispensed. The very fact that the iron and quinine citrate is slowly soluble renders it preferable to the soluble iron and quinine citrate, because the more slowly soluble a bitter compound is, the less does it affect the palate of the patient.

It will be noticed that this official compound is directed to contain 11.5 per cent. of dried quinine, and that a process of assaying the same is given in the pharmacopœia. An assay of the amount of iron is also given in the pharmacopœia, which directs that it contain 13.5 per cent. of the metal.

Iron and quinine citrate, like all the scale salts, is a tonic.

Dose.—250 milligrammes (4 grains).

FERRI ET QUININÆ CITRAS SOLUBILIS. Soluble Iron and Quinine Citrate.

It should contain not less than 11.5 per cent. of dried quinine, and ferric citrate corresponding in amount to not less than 13.5 per cent. of metallic iron. It should be kept in well-stoppered bottles, protected from light.

Thin, transparent scales, of a greenish, golden-yellow color, without odor, and having a bitter, mildly ferruginous taste; deliquescent.

Rapidly and completely soluble in cold water, partially soluble in alcohol.

When strongly heated the salt chars, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction upon red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

An aqueous solution of the salt shows a slightly acid reaction with blue litmus-paper.

Tests for Identity.—See Iron, Ammonia, and Citrates in Part V. See Quinine under Citrate of Iron and Quinine.

Impurities.—Alkaline citrates and tartrates, given above. Tartrates. Details in Part V.

Assay.—As in iron and quinine citrate.

Remarks.—This official represents citrate of iron and quinine, which has been rendered more soluble by the use of water of ammonia. It is a constituent of *bitter wine of iron* (p. 258), and there is little need of saying more concerning this compound, other than has been mentioned in discussing the previous official, being used for the same purposes.

Dose.—250 milligrammes (4 grains).

FERRI ET STRYCHNINÆ CITRAS. Iron and Strychnine Citrate.

It should contain not less than 0.9 nor more than 1 per cent. of strychnine, and ferric citrate corresponding in amount to not less than 16 per cent. of metallic iron. It should be kept in well-stoppered bottles, protected from light.

Thin, transparent scales, varying in color from garnet-red to yellowish-brown, without odor, and having a bitter, slightly ferruginous taste; deliquescent in moist air.

Readily and completely soluble in water, partially soluble in alcohol.

When strongly heated the salt chars, and finally leaves a residue of ferric oxide, which, when moistened with hot water, should not show an alkaline reaction with red litmus-paper (absence of *citrates* or *tartrates* of the *alkali metals*).

The aqueous solution of Iron and Strychnine Citrate is slightly acid to litmus-paper, and is not immediately precipitated, but rendered darker in color, by ammonia water.

Tests for Identity.—See Iron and Citrates in Part V. *Test for strychnine*, see assay (Part V).

Impurities.—Alkaline citrates and tartrates, given above.

Assay.—Details in Part V.

Remarks.—This scale salt is made by adding a solution of the alkaloid strychnine, in citric acid, to a solution of iron and ammonium citrate.

It is important to note that in making the iron and quinine citrate and iron and strychnine citrate, the uncombined alkaloid is used with the citric acid, and not one of its salts. It will be seen that this preparation contains about 1 per cent, of strychnine and 16 per cent. of iron. The assays of both these constituents are provided in the pharmacopœia.

By reason of containing the poison strychnine the dose of iron and strychnine citrate is somewhat less than that of the other scale salts.

Dose.—125 milligrammes (2 grains).

FERRI PHOSPHAS SOLUBILIS. Soluble Ferric Phosphate.

It should contain Ferric Phosphate corresponding in amount to not less than 12 per cent. of metallic iron, and should be kept in amber-colored, well-stoppered bottles, protected from light.

Thin, bright green, transparent scales, without odor, and having an acidulous, slightly saline taste. The salt is permanent in dry air when excluded from light, but when unprotected soon becomes dark and discolored.

Freely and completely soluble in water; insoluble in alcohol.

The aqueous solution of the salt shows a slightly acid reaction with blue litmus-paper. The addition of ammonia water to the solution of the salt produces a reddish-brown color.

Tests for Identity.—See Iron and Phosphates in Part V.

Assay.—Details in Part V.

FERRI PYROPHOSPHAS SOLUBILIS. Soluble Ferric Pyrophosphate.

It should contain Ferric Pyrophosphate corresponding in amount to not less than 10 per cent. of metallic iron, and should be kept in amber-colored, well-stoppered bottles, protected from light.

Thin, apple-green, transparent scales, without odor, and having an acidulous, slightly saline taste. The salt is permanent in dry air when excluded from light, but when unprotected soon becomes discolored.

Freely and completely soluble in water; insoluble in alcohol. The aqueous solution of the salt shows a slightly acid reaction with blue litmus-paper.

Tests for Identity.—See Iron and Pyrophosphates in Part V.

Assay.—Details in Part V.

Remarks.—Since these two salts closely resemble each other, they can be discussed together.

The present pharmacopœia omits the recipe for manufacture of these two salts, but the pharmacopœia of 1890 directed that they be made by mixing solution of ferric citrate with a solution of sodium phosphate (or of sodium pyrophosphate) and scaling the resulting fluid.

Both ferric phosphate and ferric pyrophosphate are, in the pure form, insoluble, but the mixed products resulting from the combination of ferric phosphate or pyrophosphate with alkaline citrates are soluble. Hence these two soluble forms are not definite compounds, but a mixture of sodium citrate with ferric phosphate or pyrophosphate, and may be called sodioferric citrophosphate and sodioferric citropyrophosphate respectively.

Both of these compounds differ from the other scale salts of iron, first, in being indefinite chemicals, and, secondly, by reason of the characteristic green color of the scales. Both are highly esteemed tonics, and are administered in about the same dose.

Dose.—Of both, 250 milligrammes (4 grains).

Soluble ferric phosphate is a constituent of three preparations of the present pharmacopœia: *glycerite of the phosphates of iron, quinine, and strychnine* (p. 207), and *elixir of the phosphates of iron, quinine, and strychnine* (p. 205), and the *syrup of the phosphates of iron, quinine, and strychnine* (p. 194).

CHAPTER XXXII

ARSENIC, ANTIMONY, TIN, GOLD, AND PLATINUM

ARSENIC

Symbol, As. Atomic weight, approximately 75

THE chief ores from which this element is derived are *realgar*, As_2S_2 , *orpiment*, As_2S_3 ; the latter word, a corruption of “auri pigmentum,” which, translated, means the color of gold or of fire, so-called because of its brilliant yellow tint. Another ore of arsenic—*flowers of arsenic* or *white arsenic*— As_2O_3 , is mined in certain districts of Styria, where the natives become habituated to its use and thus are immune from its poisonous action.

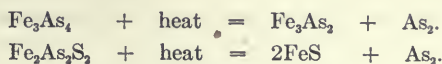
Arsenic has been known from comparatively early times. The red and yellow sulphides were known to the Greek writers. The white arsenic appears to have been known to Geber in the eighth century. In the eleventh century Avicenna differentiated between the white arsenic and the red and yellow. Albertus Magnus, in the thirteenth century, appears to have isolated the element, and the practical separation was a matter of technic in the seventeenth century.

The word arsenic is derived from the Greek word *arsenikon*, which was the term used to designate the ore orpiment. The *metallurgy* of arsenic is somewhat similar to that of the other elements which we have considered, it being isolated by heating the oxide with coal, as shown in the following equation:



This process is not used much now, since native arsenic oxide is comparatively rare, and most of the element is made by heating

arsenical iron, and from mispickel, when arsenic is freed and sublimes. The respective reactions are:

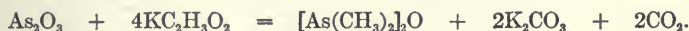


Metallic arsenic occurs in the form of crystalline masses, which are steel gray when freshly sublimed, but becoming dull and black on exposure to air. Besides this crystalline form, amorphous forms are known.

Arsenic and antimony, like zinc and aluminum, form a group about midway between the metals and the non-metals, and the non-metallic character of arsenic and antimony is even more strikingly demonstrated than in the case of zinc and aluminum. In fact, the most abundant compound of arsenic is one in which arsenic shows its negative character, namely, in arsenic trioxide (arsenous acid, U. S. P. 1890). But, while showing many points in common with the non-metallic elements, its metallic properties are well defined. Thus, it is precipitated with hydrogen sulphide, as are the other metals of this group, and combines with acids to form the salts of arsenic, such as arsenic chloride and arsenic iodide.

On the other hand, when combined with alkalis, the negative side is clearly demonstrated, and among the official compounds we find solution of potassium arsenate and sodium arsenate.

The **tests** for arsenic are as follows: The yellow sulphide (orpiment), produced when hydrogen sulphide is passed through an acidulated solution of arsenous acid. Silver nitrate test solution produces a yellow precipitate of silver arsenite or a brown precipitate of silver arsenate, according to the valence of the arsenic in the suspected solution. A characteristic test for arsenic is the so-called *cacodyl reaction*, which is brought about by fusing the arsenic with potassium or sodium acetate, when a disgusting odor of cacodyl is produced; for details descriptive of this organic compound, see p. 672. The equation is:



Compounds of arsenic, treated with copper sulphate and ammonia, yield a characteristic green precipitate of copper arsenite. This green insoluble substance was formerly largely used in the technic under the name of *Scheele's green*, as a coloring-matter. The danger of this body, however, has caused it to fall more or less into disuse.

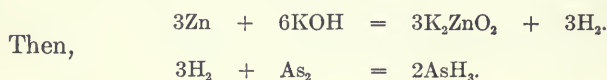
Arsenic has formerly largely employed by poisoners, and accordingly many tests have been devised for the detection of the element, such as the tests suggested by Marsh, Fleitmann, Gutzeit, and Reinsch. The principle of action of the first three of these is based on the formation of the gas, hydrogen arsenide, AsH_3 , which is produced by the union of hydrogen (generated by action of sulphuric acid on zinc), with arsenic in the suspected substance, forming hydrogen arsenite, AsH_3 , which is analogous to ammonia, NH_3 ; to phosphine (phosphoretted hydrogen, PH_3), that spontaneously combustible gas mentioned on p. 512; and to antimoniuiretted hydrogen (stibine, SbH_3),

mentioned a little further along. These four gases can best be compared in tabulated form:

Nitrogen,	valence III	gives ammonia, NH_3 ,	graphically	$\begin{array}{c} \text{H} \\ \\ \text{N} \\ \\ \text{H} \end{array}$
Phosphorus,	“ III	“ phosphine, PH_3 ,	“	$\begin{array}{c} \text{H} \\ \\ \text{P} \\ \\ \text{H} \end{array}$
Arsenic,	“ III	“ arsine, AsH_3 ,	“	$\begin{array}{c} \text{H} \\ \\ \text{As} \\ \\ \text{H} \end{array}$
Antimony,	“ III	“ stibine, SbH_3 ,	“	$\begin{array}{c} \text{H} \\ \\ \text{Sb} \\ \\ \text{H} \end{array}$

Hydrogen burns to pure water ($\text{H}_2 + \text{O} = \text{H}_2\text{O}$), while AsH_3 , in burning, yields water and metallic arsenic ($2\text{AsH}_3 + 3\text{O} = 3\text{H}_2\text{O} + \text{As}_2$); the latter separates as an arsenic spot on a piece of porcelain, just as the carbon in gas separates as soot when burned in a fish-tail burner. The details of Marsh's test is beyond the limits of this work, and the reader is referred to some book on analytic chemistry. (See Preface.)

Mention, however, must be made of the fact that antimony compounds treated with nascent hydrogen produced from zinc and sulphuric acid form antimoniuired hydrogen, SbH_3 , which, on burning, likewise deposits a stain on a porcelain dish. This latter stain is, however, brown black, and is not soluble in Labarraque's solution, as is the arsenic spot. Marsh's test is no longer given in the pharmacopœia, being replaced by a modification which does away with the cumbersome apparatus used in Marsh's test. This modification is *Gutzeit's test*, in which the hydrogen is developed from zinc and sulphuric acid in a test-tube, and the suspected substance added. If arseniuired hydrogen forms, it blackens the cap of filter-paper moistened with silver nitrate solution, covering the open end of the tube. Like Marsh's test, this responds when antimony is present, and, therefore, by far the best test is that of *Fleitmann*, in which the hydrogen is not developed by action of acid on zinc, but by treating zinc with potassa, the reaction being:



In this reaction antimony will not take part, even if present, and therefore nothing but arsenic can be detected by this means. The detection of the arsenic in this case is identical with that in *Gutzeit's test*, by the blackening of silver nitrate.

Reinsch's test is produced by placing a sheet of bright metallic copper into the suspected solution. If arsenic be present, an electrolytic action takes place, with the deposition of that element on the copper plate, which accordingly becomes blackened. *Bettendorf's test* consists in dissolving arsenic in concentrated hydrochloric acid, adding solution of stannous chloride, and introducing into the test-tube containing this mixture a piece of tin-foil. If arsenic be present, a brown coloration takes place.

In experimenting with Marsh's test, the operator should be careful regarding the inhaling of the vapors produced, as the hydrogen arsenide vapors are very poisonous, the chemist *Gehre* having been

killed by this substance during experiments in 1815. It has been noted that wall-paper colored green by the use of Scheele's green produces symptoms of arsenical poisoning in persons in the room. The exact reason for this is not clearly defined, but at the present time it is considered to be due to the formation of AsH_3 , through the action of moisture on the walls with the arsenical color and inhalation of the gas by the occupants of the room.

Arsenic, like nitrogen and phosphorus, exhibits the valence III and V, as shown in two oxides,— As_2O_3 and As_2O_5 ,—which are called arsenous and arsenic oxides respectively.

The official preparations of arsenic are:

Arsenic trioxide. At least 99.8 per cent. absolute As_2O_3 .

Solution of arsenous acid, containing 1 per cent. arsenic trioxide.

Solution of potassium arsenite, containing 1 per cent. arsenic trioxide.

Sodium arsenate. At least 98 per cent. absolute $\text{Na}_2\text{HAsO}_4 + 7\text{H}_2\text{O}$.

Exsiccated sodium arsenate. At least 98 per cent. absolute Na_2HAsO_4 .

Solution of sodium arsenate, containing 1 per cent. exsiccated sodium arsenate.

Arsenous iodide, presumably AsI_3 , at least 82.7 per cent. iodine and 16.3 per cent. metallic arsenic.

Solution of arsenous and mercuric iodides, containing 1 per cent. arsenous iodide and 1 per cent. mercuric iodide.

ARSENI TRIOXIDUM. Arsenic Trioxide.

$$\text{As}_2\text{O}_3 = 196.44$$

[ACIDUM ARSENOSUM, PHARM. 1890]

It should contain not less than 99.8 per cent. of pure Arsenic Trioxide.

A heavy solid occurring either as an opaque, white powder, or in irregular masses of two varieties: one, amorphous, transparent, and colorless, like glass; the other, crystalline, opaque, and white, resembling porcelain. Frequently the same piece has an opaque, white, outer crust enclosing the glassy variety. Contact with moist air gradually changes the glassy into the white, opaque variety. Both are odorless and tasteless.

In cold water both varieties dissolve very slowly, the degree of solubility varying according to conditions and time, the glassy variety requiring about 30, the porcelain-like or crystalline powder about 100 parts of water at 25°C . (77°F). Both are slowly but completely soluble in 15 parts of boiling water. In alcohol, Arsenic Trioxide is but sparingly soluble, but it is soluble in about 5 parts of glycerin. Oil of turpentine dissolves only the glassy variety. Both varieties are freely soluble in hydrochloric acid and in solutions of alkali hydroxides and carbonates.

When slowly heated in a test-tube, Arsenic Trioxide yields a sublimate of minute, brilliant, transparent, octahedral crystals. When heated rapidly to about 200°C . (392°F .) the amorphous variety fuses, then sublimes, while the crystalline variety sublimes without fusing; no residue should remain after sublimation. When covered with charcoal in an ignition-tube, and strongly heated, Arsenic Trioxide is deoxidized, and metallic arsenic is deposited on the cooler portion of the tube as a mirror having a metallic lustre.

Cupric ammonium sulphate T.S. produces in an aqueous solution a bright green precipitate.

If the green precipitate be dissolved in ammonia water, a deep-blue colored solution should be produced.

An aqueous solution of Arsenic Trioxide has a faintly acid reaction upon blue litmus-paper.

Silver ammonium nitrate T.S. produces in an aqueous solution a lemon-yellow precipitate, which dissolves on the addition of ammonia water; when this solution is heated, metallic silver is deposited (distinction from *arsenic acid*).

Hydrogen sulphide T.S. colors a solution of Arsenic Trioxide yellow; if a few drops of hydrochloric acid be added, it precipitates lemon-yellow arsenic trisulphide, which should be completely soluble in ammonium carbonate T.S. (absence of *antimony*, *tin*, and *cadmium*).

When Arsenic Trioxide is carefully heated in a dry test-tube of hard glass, it should sublime without leaving a residue, and the sublimate should not at first show a yellow color (absence of *non-volatile matter* and of *arsenous sulphide*).

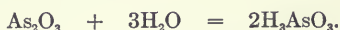
If 1 Gm. of Arsenic Trioxide be dissolved in 10 Cc. of ammonia water, with the aid of a gentle heat, a colorless solution should be produced.

Tests for Identity.—Given above.

Impurities.—Arsenic acid, antimony, tin, cadmium, arsenous sulphide, non-volatile matter. Details given above.

Assay.—Details in Part V.

Remarks.—This most important of the official arsenical preparations, called arsenous acid by the pharmacopœia of 1890, is more properly termed arsenic trioxide by U. S. P. VIII., since it is the anhydride As_2O_3 , the true acid being produced only when the official substance is dissolved in water:



Hence the aqueous solution still retains as official name the phrase, *liquor acidi arsenosi*.

Arsenic trioxide is prepared by the roasting of arsenical ores, when the oxide which is formed sublimes and is caught in appropriate condensing chambers. It is then purified by resublimation.

Arsenic trioxide acts as a reducing agent, being oxidized by potassium permanganate, and converting iodine into hydriodic acid. This explains the volumetric estimation of the chemical by means of a decinormal iodine solution. (See p. 1004.) A volumetric estimation of the chemical can also be effected by use of decinormal permanganate solution.

Arsenous acid is a valuable alterative. Applied in the form of a powder, it acts as an escharotic, and for this purpose was formerly employed for the dusting of cancerous growths. Its action in this way, however, was dangerous, and at present it is now rarely so used.

Many of the largely advertised rat-poisons consist of arsenous oxide, either free or admixed. The antidote for such nostrums is, therefore, the same as that for arsenous oxide, namely, ferric hydrate or ferric hydrate with magnesia. (See p. 564.)

Dose.—2 milligrammes ($\frac{1}{80}$ grain).

LIQUOR ACIDI ARSENOSI. Solution of Arsenous Acid.

For recipe see p. 183.

This is a 1 per cent. aqueous solution of arsenic trioxide, containing a sufficient quantity of hydrochloric acid to insure a complete solution. In the Pharmacopœia of 1870 it was styled the *solution of arsenous chloride*, but investigations have shown that the hydrochloric acid, in this case, acts merely as a solvent, not converting the oxide into chloride.

Dose.—0.2 Cc. (3 minims).

LIQUOR POTASSII ARSENITIS. Solution of Potassium Arsenite.

An aqueous solution, which should contain Potassium Arsenite corresponding in amount to 1 per cent. of arsenic trioxide.

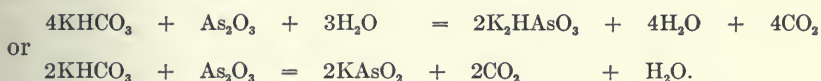
<i>Recipe</i> .—Arsenic Trioxide, in fine powder, ten grammes....	10 Gm.
Potassium Bicarbonate, twenty grammes.....	20 Gm.
Compound Tincture of Lavender, thirty grammes..	30 Gm.
Distilled Water, a sufficient quantity,	
To make one thousand grammes..	1000 Gm.

Boil the Arsenic Trioxide and Potassium Bicarbonate, in a tared dish, with one hundred grammes of Distilled Water, until solution has been effected. Then add enough Distilled Water to make the solution weigh nine hundred and seventy grammes, and, lastly, add the Compound Tincture of Lavender. Filter through paper.

Assay.—Details in Part V.

Remarks.—This preparation, commonly called “Fowler’s solution,” is made from arsenic trioxide and potassium bicarbonate, colored with compound tincture of lavender. It contains 1 per cent. of arsenic trioxide.

The equation for its manufacture is either—

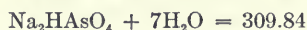


The pharmacopœia directs that the potassium bicarbonate and the arsenic trioxide be boiled together in water to insure chemical combination. If a solution of potassium bicarbonate and a solution of arsenic trioxide are mixed together in the cold, no reaction takes place, and, in fact, some authorities claim that even boiling produces but a slight effect on the two constituents. How little decomposition occurs is shown by the fact even when twice the amount of arsenous acid is boiled with solution of potassium bicarbonate, and even when such solution is boiled to dryness and then redissolved in water, the latter liquid still effervesces with acids.

Despite this fact, some authorities claim that a meta-arsenite of potassium (KAsO_2) is produced; while others hold to the ortho-arsenite formula, K_2HASO_3 ; this is supposedly the acid potassium salt of H_3AsO_3 . Fowler’s solution is one of the most popular of arsenical preparations, being particularly esteemed as an alterative in cases of malaria. Many physicians in malarial countries consider this preparation almost in the light of a specific.

Dose.—0.2 Cc. (3 minims).

SODII ARSENAS. Sodium Arsenate.



It should contain in an uneffloresced condition not less than 98 per cent. of pure Di-sodium-ortho-arsenate [$\text{AsO}(\text{OH})(\text{ONa})_2 + 7\text{H}_2\text{O}$] and should be kept in well-stoppered bottles.

Colorless, transparent, monoclinic prisms, odorless, and having a mild, alkaline taste; *caution should be used in tasting this salt, as it is very poisonous*. Efflorescent in dry air and somewhat deliquescent in moist air.

Soluble in 1.2 parts of water at 25° C. (77° F.) and very soluble in boiling water; very sparingly soluble in cold, but nearly insoluble in boiling alcohol. When gently heated, the salt loses 5 molecules of water (28.8 per cent.) and is converted into a white powder. At 148° C. (298.4° F.) it loses all of its water of crystallization, at a higher temperature the salt fuses, and at a red heat it is converted into pyroarsenate.

Sodium Arsenate should respond to the tests of identity and purity prescribed under *Sodii Arsenas Exsiccatus*.

Tests for Identity.—See Exsiccated Sodium Arsenate.

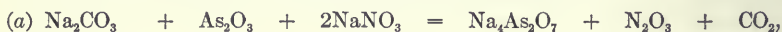
Remarks.—Sodium arsenate is made by adding arsenous acid to the mixture of sodium carbonate and sodium nitrate, and heating same to redness and pouring out the fused mass on a slab, and treating it with boiling water as soon as solidified. The solution is then evaporated until crystallization takes place; the crystals are removed from the mother liquor and dried according to the usual method.

This process furnishes an interesting example of oxidation. Arsenic has the valence III and V, and the two oxides, As_2O_3 and As_2O_5 , when treated with water, yield respectively the true arsenous acid (H_3AsO_3) and arsenic acid (H_3AsO_4), the latter being analogous to phosphoric acid, H_3PO_4 . (See p. 373.) An arsenate is a salt of arsenic acid, and if we treat sodium carbonate with arsenous acid (which is common, while arsenic acid is rare), we would not get an arsenate, but an arsenite, just as we did in Fowler's solution.

It will be noticed that the arsenical ingredient used above is arsenous oxide (As_2O_3). We have already learned (p. 374) that "ous" acids give "ite" salts, a proof of which, with the very acid we are now considering, is shown in Fowler's solution,—liquor potassii arsenitis,—where we see that arsenous acid, cooked with potassium bicarbonate, yields potassium arsenite—not the arsenate—shown in equation "a" given on p. 577.

In exactly similar manner sodium carbonate and arsenous acid yield Na_2HAsO_3 , sodium arsenite. Necessary to form the arsenate, is arsenic acid, As_2O_5 or H_3AsO_4 , and, accordingly, if we start out with arsenous acid, it must be oxidized to arsenic acid before the arsenate can be formed. It may be recalled that in making sulphuric acid, sulphurous oxide, SO_2 , was oxidized to sulphuric oxide, SO_3 , and that the oxidation was accomplished by nitric acid. In oxidizing arsenous oxide to arsenic oxide in the manufacture of sodium arsenate nitric acid is not adapted, since it, rather than the arsenic preparation, would react with the sodium carbonate, the final product being a mixture of sodium nitrate and arsenic acid; hence we employ sodium nitrate, NaNO_3 , which at a high heat splits in a manner akin to nitric acid itself, with the liberation of oxygen.

The usually accepted equation of manufacture—

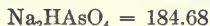


is more complex than the explanation just given, because of the presence of an intermediate product, sodium pyroarsenate, $\text{Na}_4\text{As}_2\text{O}_7$. This body, however, is analogous to sodium pyrophosphate, $\text{Na}_4\text{P}_2\text{O}_7$, the latter a salt of pyrophosphoric acid, $\text{H}_4\text{P}_2\text{O}_7$, the chemistry of which was explained on p. 473.

Sodium arsenate is used as an alterative and tonic.

Dose.—5 milligrammes ($\frac{1}{10}$ grain).

SODII ARSENAS EXSICCATUS. Exsiccated Sodium Arsenate.



It should contain not less than 98 per cent. of pure anhydrous Di-sodium-ortho-arsenate $[\text{AsO}(\text{OH})(\text{ONa})_2]$.

Recipe.—Sodium Arsenate Crystals, a sufficient quantity.

Break the crystals into small fragments, and allow them to effloresce at a temperature between 40° and 50° C. (104° and 122° F.) until they are completely disintegrated; then gradually increase the temperature to 150° C. (302° F.), and continue the drying until the product ceases to lose weight. Reduce it to a fine powder, and transfer it to dry, well-stoppered bottles.

An amorphous, white powder; odorless and having a mildly alkaline taste; *it should be tasted with great caution, as the salt is very poisonous.* Permanent in dry air.

Soluble in 3 parts of water at 25° C. (77° F.) and very soluble in boiling water; very sparingly soluble in cold, but nearly insoluble in boiling alcohol. When heated to 150° C. (302° F.) the salt should not lose weight; at red heat it is converted into pyroarsenate. It imparts an intense yellow color to a non-luminous flame.

Tests for Identity.—See Sodium and Arsenates in Part V.

Impurities.—Arsenite, lead, copper, and iron. Details in Part V.

Remarks.—This new official is made by driving off the seven molecules of water of crystallization from the crystalline sodium arsenate by use of a temperature of 150° C. (302° F.).

It is used for making the official *solution of sodium arsenate*. While the exsiccated salt was not recognized by the Pharmacopœia of 1890, the solution of sodium arsenate of that pharmacopœia was prepared by first exsiccating the crystallized salt, experience showing that when that solution is prepared from the crystalline salt, it is of uncertain strength, since the crystals are usually more or less efflorescent.

Dose.—3 milligrammes ($\frac{1}{20}$ grain).

Liquor Sodii Arsenatis.—For recipe see p. 187.

This official is a 1 per cent. solution of exsiccated sodium arsenate in water.

Dose.—0.2 Cc. (3 minims).

ARSENI IODIDUM. Arsenous Iodide.



It should contain not less than 82.7 per cent. of iodine, and 16.3 per cent. of metallic arsenic. Arsenous Iodide should be kept in amber-colored, glass-stoppered vials, in a cool place, carefully protected from the light.

An orange-red, inodorous, crystalline powder, stable when protected from direct sunlight and kept in a cool place.

Soluble, with partial decomposition, in about 12 parts of water and in about 28 parts of alcohol at 25° C. (77° F.); completely soluble in chloroform, carbon disulphide, or ether.

No loss of iodine occurs when Arsenous Iodide is heated upon a water-bath, but at higher temperatures it completely volatilizes. When warmed with a few drops of nitric acid, brown vapors of nitrous oxide are evolved, followed by violet vapors of iodine.

The aqueous solution has a yellow color, is neutral to litmus-paper, and upon standing gradually decomposes into arsenous and hydriodic acids.

If hydrogen sulphide T.S. be added to an aqueous solution of Arsenous Iodide, acidulated with hydrochloric acid, a lemon-yellow precipitate of arsenous sulphide is produced.

Tests for Identity.—Given above.

Assay.—Details in Part V.

Remarks.—This compound was formerly made by fusing molecular quantities of metallic arsenic and iodine into a test-tube and pouring the fused mass on a slab in order to permit it to solidify. This process, however, was very wasteful of the iodine, and therefore a much better method is by making a solution of iodine in carbon disulphide, and sprinkling therein finely powdered arsenic until the purple color is discharged.

Arsenic iodide is a comparatively unstable compound, losing its iodine by volatilization until its composition is very largely changed. It is, therefore, at its best a rather uncertain preparation, as is the case with sulphur iodide.

This substance is used as an alterative.

Dose.—5 milligrammes ($\frac{1}{10}$ grain).

LIQUOR ARSENI ET HYDRARGYRI IODIDI.

For recipe see p. 184.

This official, commonly called *Donovan's solution*, after the originator, contains 1 per cent. of arsenous iodide and 1 per cent. of mercuric iodide.

The solution should be practically colorless. On standing, it assumes a reddish tint, due to the separation of free iodine, and such a reddened Donovan's solution should not be dispensed. The change can be largely obviated by adding a globule of mercury to the solution. It is hardly necessary to say that care must be taken to prevent the pouring out of the mercury globule with the dispensed solution.

Donovan's solution is used as an alterative and tonic.

Dose.—0.1 Cc. ($1\frac{1}{2}$ minims).

It will be seen that the pharmacopœia recognizes four solutions containing arsenic compounds (see p. 187), and that, while all these are 1 per cent. preparations, all *do not* contain 1 per cent. of arsenic trioxide.

ANTIMONY

Symbol, Sb. Atomic weight, approximately 122

This element is sometimes found native in uncombined form, but its chief ore is the sulphide,— Sb_2S_3 ,—which is called stibnite.

The symbol Sb is applied to antimony, is derived from the Latin name of the element, *stibium*, which is in turn derived from the Greek word *stibi*, which was the name applied to that native sulphide, which has been known since classic times. The etymology of the word antimony has been explained by a curious fable, which relates that Basel Valentine, a monk of the monastery of Erfurt, experimented with the salts of this element on his brother monks, with fatal results, whereupon he named the element *antimoine*—against monks. This theory of the origin of the name, however, has been exploded since the word antimonium was used in a book of Constantine of Salerio (A. D. 1100), and merciless critics have even denied the existence of such a man as Basel Valentine. Suffice it to say that there appeared during the fifteenth century a book entitled "The Triumphant Chariot of Antimony," purported to have been printed from the manuscript of this monk Basel Valentine. It was through the publication of this work that the use of antimony in medicine became popularized. In the sixteenth and seventeenth centuries antimony and its compounds were among the most popular of medicines, the element being isolated by Boerhave. One of the applications of this substance was in the form of a cup made of this metal (*pocula emetica*). Acid or wine was allowed to stand in this cup until a certain amount of this metal had been dissolved. The wine, when drank, then acted as an emetic. Under the name of "*pilula perpetua*" small globules of the

metal were used in the seventeenth century, enough of the metal being dissolved from the swallowed pill by the acid juices of the stomach to produce the desired effect. The pill, voided either by emesis or through the bowels, was then saved for the next need.

The metallurgy of antimony is accomplished by heating the sulphide with metallic iron, as shown in the following equation:



In order to facilitate the separation of the metal from the ferrous sulphide a mixture of exsiccated sodium sulphate and coal is added for the purpose of forming a slag with the ferrous sulphide.

Antimony is a brilliant brittle metal, of lamellated texture, and silvery white when chemically pure. The commercial antimony, however, is usually of a bluish-white tint. Antimony is a constituent of several valuable alloys. Thus, *type-metal* is a combination of 15 per cent. of antimony and 85 per cent. of lead; while *pewter* consists of 10 per cent. of antimony, 86 per cent. of tin, 3 per cent. of zinc, and 1 per cent. of copper.

The tests for antimony are as follows: hydrogen sulphide precipitates from acid solutions of antimony golden yellow (orange) antimony sulphide (Sb_2S_3), which, like the sulphides of arsenic, tin, gold, and platinum, will dissolve in ammonium sulphhydrate solution. If, into an acid solution of antimony is placed zinc and platinum in the form of plates, an electrolytic action takes place with the deposition of antimony on the platinum as a black spot. This black spot is soluble in a solution of potassium permanganate.

As mentioned under Arsenic, Marsh's tests can be used for the detection of antimony. In fact, the two elements act so similarly when subjected to Marsh's test as to render it difficult to distinguish between them. When antimony is used in Marsh's test, however, the hydrogen flame has a tint which an expert can easily distinguish from arsenic. The spot produced by placing a clean piece of porcelain into the flame (which is none other than separated metallic antimony) is darker than the similar arsenic spot, and possesses less metallic lustre. Moreover, it is insoluble in Labarraque's solution, while the arsenic spot dissolves readily in this reagent.

Fleitmann's test does not respond to antimony, and is, therefore, to be preferred in the detection of arsenic.

Three oxides of antimony are known, the antimonous oxide, Sb_2O_3 , antimonic oxide, Sb_2O_5 , and the third oxide, Sb_2O_4 .

Sb_2O_4 can be considered as Sb_2O_3 plus Sb_2O_5 , which would give us Sb_4O_8 or $2\text{Sb}_2\text{O}_4$. Besides, it can be regarded as antimonyl antimonate ($\text{SbO})\text{SbO}_3$, to understand which the acid forms of antimony must be explained.

Already we have seen that nitrogen with valences III and V give two oxides, N_2O_3 (nitrous) and N_2O_5 (nitric). Just so are the oxides of antimony, Sb_2O_3 and Sb_2O_5 .

The nitrogen oxides, as shown on p. 369, yield, when added to water, nitrous and nitric acids respectively.

Exactly so Sb_2O_3 yields antimonous acid (HSbO_2) and Sb_2O_5 gives antimonic acid (HSbO_3). Antimonic acid will give salts, such as

potassium antimonate (KSbO_3), and in Sb_2O_4 we have antimonyl antimonate— $(\text{SbO})\text{SbO}_3$, antimonyl being a radicle of valence 1

$(\text{SbO})^1$, graphically $\text{Sb} \begin{smallmatrix} \text{=O} \\ \text{---} \end{smallmatrix}$, just as ammonium is a radicle with valence 1

$(\text{NH}_4)^1$, graphically $\text{N} \begin{smallmatrix} \text{---N} \\ \text{---N} \\ \text{---N} \end{smallmatrix}$.

The "antimonyl," just spoken of, is found in one of the official preparations—tartar emetic.

The only preparations of antimony recognized in the present pharmacopœia are:

Antimony and potassium tartrate. At least 99.5 per cent. absolute $2\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 + \text{H}_2\text{O}$.

Wine of antimony, containing 0.4 per cent. antimony and potassium tartrate.

Compound syrup of squill, containing 0.2 per cent. antimony and potassium tartrate.

The pharmacopœia of 1890 also recognized—

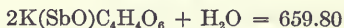
Antimony sulphide.

Purified antimony sulphide.

Sulphurated antimony.

Antimony oxide.

ANTIMONII ET POTASSII TARTRAS. Antimony and Potassium Tartrate.



It should contain not less than 99.5 per cent. of pure Antimony and Potassium Tartrate [$2\text{C}_2\text{H}_2(\text{OH})_2(\text{COOK})\text{COOSbO} + \text{H}_2\text{O}$].

Colorless, transparent crystals of the rhombic system, becoming opaque and white on exposure to air, or a white, granular powder; without odor and having a sweet, afterward disagreeable, metallic taste.

Soluble in 15.5 parts of water at 25°C . (77°F .) and in 3 parts of boiling water, but insoluble in alcohol, which precipitates it from its aqueous solution in the form of a crystalline powder.

When heated to 110°C . (230°F .) the salt loses its water of crystallization (2.71 per cent.). When heated to redness it chars, emits an odor resembling that of burning sugar, and leaves a blackened residue having an alkaline reaction.

On adding sodium carbonate T.S. to crushed crystals of the salt, effervescence should not occur (absence of *potassium bitartrate*).

Tests for Identity.—See Antimony, Potassium, and Tartrates in Part V.

Impurities.—Sulphates, chlorides, calcium, iron, heavy metals, arsenic. Details in Part V. Potassium bitartrate. Details given above.

Assay.—Details in Part V.

This official, commonly called *tartar emetic*, is, in truth, antimonyl and potassium tartrate, since the radicle $(\text{SbO})^1$ enters the compound rather than the element itself. Tartaric acid has the power of easily forming double salts. (See p. 378.) Rochelle salt was one of these double salts, and "tartar emetic" differs from Rochelle salt only in the substitution of antimonyl (SbO) for sodium:



It is made by boiling the antimony oxide with potassium bitartrate and water for an hour. When so treated, the hydrogen of the potassium bitartrate is eventually replaced by the antimonyl group furnished by the antimony oxide.

Antimony and potassium tartrate, as its synonym would indicate, is used as an emetic, in large doses, while in small doses it acts as an alterative and diaphoretic and expectorant. It was formerly largely used as an emetic, but its action was so violent that it has largely fallen into disuse. If given in an overdose, the antidote is tannin.

Dose.—Expectorant, 5 milligrammes ($\frac{1}{10}$ grain). Emetic, 30 milligrammes ($\frac{1}{2}$ grain).

Tartar emetic is a constituent of two official galenics—*wine of antimony* (p. 257), containing 0.4 per cent. of tartar emetic dissolved in boiling water and added to white wine, and *compound syrup of squill* (p. 200), containing 0.2 per cent. of tartar emetic combined with a fluidextract of senega, fluidextract of squill, and syrup.

ANTIMONII OXIDUM (U. S. P. 1890). Antimony Oxide.

Shows that element with the valence III, it being the "ous" oxide, Sb_2O_3 . It was the antimony ingredient of the *antimonial powder* (U. S. P. 1890).

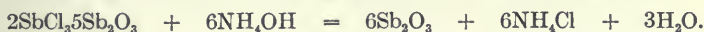
The manufacture of this compound is rather intricate. Antimony sulphide is first dissolved in hydrochloric acid, whereby soluble antimony chloride, *butter of antimony*, is produced:



The filtered solution is then poured into a large quantity of water, with the production of an insoluble oxychloride, shown by the following equation:



This insoluble oxychloride (*powder of algaroth*) is then treated with ammonia water, which converts it into the pure oxide, as is shown in the following equation:



This oxide is washed to free it from ammonium chloride and then dried.

The product is a white powder, insoluble in water.

Antimony oxide is used as an alterative in doses of 0.05 Gm.

Antimonial powder (U. S. P. 1890) or *James' powder* is a galenic made by triturating 33 Gm. of the oxide with 67 Gm. of calcium phosphate. (See p. 301.)

Antimony sulphide was official in three forms in the pharmacopœia of 1890.

First: The crude ore containing arsenic, despite the efforts to remove it by fusion.

Second: The purified, from which the arsenous sulphide has been removed by water of ammonia.

Third: Sulphurated, which is made by a process similar to that followed in making precipitated sulphur. (See p. 399.) Although the chemical reaction is quite different in both cases, the substance which we desire to purify, and which is insoluble, is dissolved by boiling with alkali (lime in the case of precipitated sulphur, soda in sulphurated antimony), and the product desired precipitated with acid. "*Kermes mineral*" (as the sulphurated antimony is called), is not a pure sulphide, Sb_2S_3 , but is a mixture of this with Sb_2O_3 . It is called *Kermes mineral*, from a supposed resemblance to *Kermes*, an ancient dye-stuff.

ANTIMONII SULPHIDUM (U. S. P. 1890). Antimony Sulphide.

This substance is too crude for internal use, containing, as it does, a large quantity of arsenic. It is used very largely in veterinary practice as an alterative under the name of *black antimony*, and should never be dispensed for human consumption.

ANTIMONII SULPHIDUM PURIFICATUM (U. S. P. 1890). Purified Antimony Sulphide.

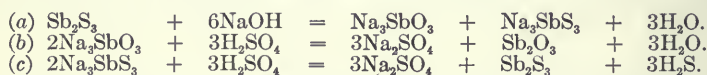
This product represented crude antimony sulphide, purified by washing with ammonia, the theory of this washing being that arsenic sulphide is soluble in ammonia water. However, antimony sulphide itself is somewhat soluble in ammonia water, and therefore the process is wasteful. Equally good results would be obtained, as far as freeing the sulphide from arsenic is concerned, by washing with a mixture of ammonia water and ammonium carbonate, in which solvent an antimony sulphide is scarcely soluble.

The purified sulphide of antimony is used as an alternative in doses of 1 grain (0.06 Gm.).

ANTIMONII SULPHURATUM (U. S. P. 1890). Sulphurated Antimony.

As just mentioned, Kermes mineral is made by boiling antimony sulphide with soda and precipitating the alkaline solution with sulphuric acid.

The equations occurring in this process are as follows:



Sulphurated antimony is an alternative and diaphoretic. It is much esteemed by French physicians, who find it a valuable constituent for cough-mixtures, a favorite recipe for this purpose being 1 grain of sulphurated antimony in 1 ounce of syrup of wild cherry, a teaspoonful being given as a dose. Its dose is usually from $\frac{1}{2}$ to 1 grain.

Compound pills of antimony, or *Plummer's pills*, consist of sulphurated antimony, powdered guaiac, and calomel. They are used in secondary syphilis, one or two pills being the dose.

Another unofficial antimony compound worth passing mention is *pyroantimonic acid*, $\text{H}_2\text{Sb}_2\text{O}_7$. This compound, as it will be seen, has the formula analogous to that of pyrophosphoric acid, already discussed on p. 473, its potassium salts being used for the detection of sodium, sodium pyroantimoniate being practically the only insoluble sodium compound.

TIN

Symbol, Sn. Atomic weight, approximately 118

While this element yields no official compounds, it is of sufficient importance to justify passing notice.

The source of tin is the oxide, SnO_2 , which is commonly called *tin stone*, and mined very largely in Cornwall, England, Malacca, and Dakota.

Tin has been used for many centuries, Pliny's *kassiteros* (or *plumbum candidum*), which he says came from the Cassiterides in the Atlantic, being our modern tin. The Latin word *stannum* is derived from the Saxon *stan*, meaning stone.

The metallurgy of tin, like that of so many other metals, is accomplished by heating together coal and the oxide, freed from sulphur and arsenic by roasting, the reaction being as follows:



Tin is a brilliant white metal, very malleable, and thus can be made out into very thin sheets, which are much used by pharmacists under the name of *tin-foil*. The so-called *tin-plate* consists of sheets of iron coated with tin, either by dipping the iron into the molten metal or by rolling the tin upon the iron plate.

Tin compounds are tested as follows: Potassium hydroxide produces a white precipitate of tin hydroxide; ammonium sulphide produces a black precipitate when a stannous salt is under treatment (stannous sulphide); a yellow precipitate of stannic sulphide, if the tin compound is of the higher valence. The special test for stannous tin is the fact that if a solution of mercuric chloride is mixed with a solution of stannous salt, the mercuric salt is reduced to metallic mercury, with a corresponding separation of the metal as a gray precipitate.

Two oxides of tin are known, stannous oxide, SnO , and the stannic oxide, SnO_2 .

In these, like in the other tin compounds, the stannous form of tin exhibits the valence II, while the stannic form shows valence IV.

No compounds of tin are official. The following, however, are of passing interest in pharmaceutical chemistry. *Stannous chloride*, SnCl_2 , is made by treating the metal with hydrochloric acid, and is used in analytic work as a reducing agent, as shown in the mercury test for stannous compounds that has just been given.

Stannic chloride, SnCl_4 , was first made by Libavius (1605) by distilling tin with mercuric chloride. When water free, it is a colorless, fuming liquid, which was formerly known as *spiritus fumans Libavii*.

Tin, like zinc and aluminum, stands midway between the positive and negative elements, and while generally acting as a metal in its compounds, it forms, with alkalis, salts of stannic acid, H_2SnO_3 . The sodium salt, Na_2SnO_3 , is used largely in calico printing under the name of "*tin salt*."

GOLD

Symbol, Au. Atomic weight, approximately 196

Gold is usually found in the native, uncombined form, being associated with either sand or quartz. It is beyond the limits of this work to go into extended consideration of the production of gold, and therefore suffice it to say that it is found in certain sections of the world, such as, for instance, California, Alaska, Australia, and South Africa.

The mining of gold is accomplished in several ways, according to the condition in which the metal is found. When found associated with sand, it is usually extracted from the beds of rivers. In this case the gold is separated from the sand by the process of washing, which can be compared to levigation. (See p. 146.) If done on a small scale, the mixture is placed in a pan, and washed rapidly with water, whereby the sand is washed out, leaving the heavier gold in the bottom of the pan. The washing is performed on a large scale by the process of "placer-washing," which consists of putting the gold-bearing sand into the upper one of a series of long troughs, each trough being slightly lower than its predecessor.

Into the top trough is run a good stream of water (usually from an adjacent mountain torrent), which, passing continuously through, gradually throws out all the sand, and, at the same time, a large quantity of the gold. The mixture of sand and gold passes to a second trough, where a considerable quantity of the gold separates out, while all the sand is washed out into the third trough. And thus the process goes on until all the sand has been removed, while practically all the gold remains in the troughs.

The crude gold is purified by the amalgamation process,—that is, it is treated with mercury,—and the amalgam then submitted to distillation, when the mercury passes over, leaving the gold in the still.

When gold is found in quartz, the gold is extracted by either of two methods.

In the chlorine process the ore is roasted to remove sulphur and arsenic, the residue mixed with water and treated with chlorine. Gold is separated from the resulting gold chloride solution by precipitation with ferrous sulphate.

In the *cyanide process* the finely ground ore is percolated with a dilute solution of potassium cyanide, which dissolves the gold as a double cyanide. The metal is separated from the percolate by electrolysis.

Gold is one of the most ancient of metals, ornaments of the metal being found in the Egyptian tombs. The Latin word for gold, *aurum*, is of classic origin, being derived from the Hebrew word *zohov*, which meant the color of fire. It is of interest to note that in many of the ancient languages the word distinguishing gold is the same word used to signify yellow.

Gold is a soft yellow metal, having the specific gravity 19.00. It is the most malleable of all metals, capable of being beaten into sheets (leaves) so thin that 280,000 of them are required to measure an inch. When beaten out so thinly, the sheets are translucent, and the light transmitted through them is of a greenish color. Gold is also a very ductile metal, although it is not capable of being drawn into wire as thin as is platinum. It is not soluble in nitric acid nor in hydrochloric acid, but it does dissolve in a mixture of the two—*aqua regia* (see p. 417), which is supposed to act on the metal, not by reason of the composition of either of the constituents, but because of the free chlorine and nitrosylchloride contained in the nitrohydrochloric acid. Because of the softness of the pure metal, gold is always employed in the form of an alloy.

The gold coins of America consist of 90 per cent. of gold and 10 per cent. of copper, while the gold used in making jewelry contains a still larger quantity of copper. The rating of the gold content of jewelry gold is not by percentage, but by a clumsy commercial standard called *karats*. Pure gold is said to be 24 karats. Hence when we speak of 14-karat gold we mean that it is $\frac{14}{24}$ gold.

The *tests* for gold are as follows: Hydrogen sulphide gives the black precipitate of gold sulphide. When a fresh solution of ferrous sulphate is added to a solution of gold salt, gold separates out, and when gold salts are treated with tin chloride in the presence of a ferric salt, a beautiful purple precipitate is formed. This precipitate, which is either $\text{SnAu}_2(\text{SnO}_3)_2$, according to Debray, a mixture of metallic gold with a stannic acid, was formerly used as a pigment under the name of *purple of Cassius*.

Gold usually exhibits the valence III, although monovalent gold is known in such compounds as AuCl and Au_2O .

The only official salt of gold is—

AURI ET SODII CHLORIDUM. Gold and Sodium Chloride.

A mixture of equal parts, by weight, of anhydrous Gold Chloride [$\text{AuCl}_3 = 301.24$] and anhydrous Sodium Chloride [$\text{NaCl} = 58.06$], representing not less than 30 per cent. of metallic gold. It should be kept in well-stoppered, amber-colored vials.

An orange-yellow powder, odorless, having a saline and metallic taste, and deliquescent when exposed to damp air.

The compound is very soluble in water and at least one-half of it should be soluble in cold alcohol.

When exposed to a red heat it is decomposed, and metallic gold is separated.

A fragment of the compound imparts a persistent, intensely yellow color to a non-luminous flame.

Its aqueous solution has a slightly acid reaction, and yields with silver nitrate T.S. a white precipitate insoluble in nitric acid and soluble in ammonia water.

On bringing a glass rod moistened with ammonia water close to a portion of Gold and Sodium Chloride, no white fumes should make their appearance (absence of *free hydrochloric acid*).

If 0.5 Gm. of Gold and Sodium Chloride be dissolved in 25 Cc. of water, in a porcelain dish, the solution made alkaline by the addition of 5 Cc. of potassium

hydroxide T.S., and, after the addition of 5 Cc. of solution of hydrogen dioxide, heated for about one hour on a water-bath, a precipitate of metallic gold will be obtained, which, when washed with water slightly acidulated with hydrochloric acid, dried, and ignited, should weigh not less than 0.15 Gm. (corresponding to at least 30 per cent. of metallic gold).

The filtrate from the precipitated gold should not be affected by hydrogen sulphide T.S., nor, after being supersaturated with ammonia water, by ammonium sulphide T.S. (absence of *metallic impurities*).

Tests for Identity.—Given above.

Impurities.—Free hydrochloric acid. Metallic impurities. Details above.

Assay.—Details in Part V.

Remarks.—Gold chloride is very deliquescent, and to lessen this disagreeable quality as much as possible the pharmacopœia directs it combined with an equal weight of “decrepitated” sodium chloride. Decrepsitated salt means that from which the interstitial water has been driven by heat. Salt, when heated, crackles, because the interstitial water forms steam which explodes with sufficient force to break the crystals.

The mixture of dried gold chloride with anhydrous common salt is official as *gold and sodium chloride*.

The gold chloride is made by dissolving gold in aqua regia. The filtered solution is then evaporated, when there crystallizes chlorauric acid, $\text{HAuCl}_4\text{H}_2\text{O}$.

If this be mixed with sodium chloride, true gold and sodium chloride, $\text{AuCl}_3\text{NaCl}2\text{H}_2\text{O}$, results. This is not the official body, which, as above stated, is made by triturating dried gold chloride with an equal quantity of dried salt.

Gold chloride is best made by treating gold with chlorine, since the product obtained by evaporation of an aqua regia solution is apt to contain aurous chloride, AuCl .

Gold and sodium chloride is used as an alternative. Of late, gold chloride and other compounds have been much vaunted as remedies for the drink habit. That the so-called gold cure is efficient cannot be denied, but whether due to the gold or to the other stimulants employed is a matter still open for discussion.

Dose.—5 milligrammes ($\frac{1}{10}$ grain).

PLATINUM

Symbol, Pt. Atomic weight, 193.3

Platinum occurs native, and is found in Mexico, Brazil, and chiefly in the Ural Mountains. The metal thus obtained is always contaminated with palladium, iridium, osmium, ruthenium, and the separation of these precious metals is a matter of considerable difficulty.

Platinum was first brought to the attention of the world by de Ulloa, in 1746, who, in his descriptions of his explorations of Mexico, referred to the metal there discovered. The word *platina* (little silver) was first applied to the metal by Scheffer in 1752.

Platinum is the most ductile of all metals, it having been drawn into wire so thin that a microscope is required to see the strand. It is one of the most difficult of the metals to be fused, and for this reason it is of great value in the making of crucibles or similar vessels to be used for high heat. (See p. 82.) It must be noted, however, that platinum utensils are by no means unaffected by any substance, experience having shown that the metal forms a fusible alloy with lead and a fusible phosphide with phosphorus; that also alkaline hydrates, sulphides, hypophosphites, and

cyanides attack the platinum, eventually eating through the same; so none of these substances should be heated in platinum dishes.

Spongy platinum is an interesting form of the metal. It is made by heating platino-ammonium chloride (see below) until all the ammonia and chlorine have been driven off, leaving a spongy lump of platinum which possesses remarkable absorptive capacity for oxygen, it being stated that one volume will absorb 250 volumes of that gas. The oxygen thus absorbed is in a condition readily to combine with other substances, notably hydrogen, and this principle was made use of in the so-called Doebereiner's lamp, which was largely used before the introduction of matches. This lamp consists of an ordinary hydrogen generator, with a piece of spongy platinum placed near the nozzle, from which the hydrogen gas would be emitted. As soon as a stream of hydrogen is placed on the spongy platinum, a chemical union between oxygen and hydrogen takes place, with a heat sufficiently intense to cause the ignition of the hydrogen, and thus producing flame.

Another interesting point connected with platinum is the fact that at red heat a sheet of platinum permits hydrogen to pass through it, no other gas being thus given transit. This phenomenon has been made use of for the removal of hydrogen from a mixture of gases.

The tests for platinum are as follows: Hydrogen sulphide produces a brown sulphide, and potassium chloride added to a solution of platinic chloride will produce the characteristic yellow crystals. These crystals have already been discussed under Potassium. A similar precipitate of platinic ammonium chloride is produced when ammonium chloride is also treated with platinic chloride.

No salts of platinum are official, but *platinic chloride*, PtCl_4 , is mentioned in the appendix to the pharmacopœia as a constituent of the test solution used for testing for the presence of potassium.

CHAPTER XXXIII

CADMIUM, COPPER, AND BISMUTH

CADMIUM

Symbol, Cd. Atomic weight, 111.6

CADMIUM is a mineral which is usually found in combination with zinc; most calamines containing cadmium. The most important cadmium minerals is *greenockite*, which is chiefly cadmium sulphide.

The name is derived from the Greek word *kademia*, which was the ancient name for calamine. The element was isolated from this ore by Strohmeyer in 1817.

Cadmium is a white metal, like tin, and like it crackles when bent; it has a higher specific gravity, however, and is more tenacious.

The tests for cadmium are the yellow sulphide, produced by treating solutions of the salts with hydrogen sulphide or with ammonium sulphide, and the white hydroxide, precipitated when the solution of the salt is treated with potassium hydroxide or ammonium hydroxide. This hydroxide is insoluble in an excess of potassa, but soluble in an excess of ammonia. Sodium carbonate precipitates cadmium salts in the form of white cadmium carbonate, which is insoluble in an excess of the reagent.

Cadmium usually exhibits the valence II, as shown in the oxide, CdO . No salts of cadmium are official.

COPPER

Symbol, Cu. Atomic weight, approximately 63

This important metal is found in nature as metallic copper; also is found in the ore *copper glance*, Cu_2S ; also as cuprous oxide, while native copper carbonate forms a beautiful green mineral, *malachite*.

Copper has been known from the most ancient times, its name, cuprum, coming from *cyprum*, the isle of Cyprus, where the ores were

first mined. The metallurgy of the copper ores depends upon the character of the ore. If the ore is chiefly copper oxide, the separation is easily effected by heating ore with carbon, as expressed in the following equation:



Sulphides, on the other hand, are first roasted with sand and some substance which will act as a flux. This action partially converts the ore into copper oxide. By heating this mixture, sulphur dioxide is liberated and copper remains, as expressed by the following equation:



Chemically pure copper is prepared by the electrolysis of copper sulphate.

Copper is a brilliant red metal, ductile and malleable. When chemically pure, it is an admirable conductor of electricity, and its use for this purpose is so extensive that the supply is scarcely equal to the demand. The metal is insoluble in either diluted hydrochloric acid or diluted sulphuric acid, except in presence of air, but it does dissolve in concentrated sulphuric acid with the formation of copper sulphate.

An important use of copper is in the formation of the well-known series of alloys. Thus *brass* is made by the fusion of three parts of copper and one part of zinc. *Bronze* represents the mixture of copper, tin, zinc, and lead. *Japanese bronze* contains gold and silver, in addition to the metals above given. *Phosphor-bronze* consists of 90 per cent. of copper, 9 per cent. of tin, and 1 per cent. of phosphorus. This is a remarkably tough and durable alloy, and is, therefore, of great value in making metallic bearings. Copper coins usually consist of 95 per cent. of copper, 4 per cent. of tin, and 1 per cent. of zinc.

The *tests* for copper are: The black sulphide produced by treatment with hydrogen sulphide or ammonium sulphide; the brown-red copper ferrocyanide formed when a solution of a copper salt is treated with potassium ferrocyanide, and the green color a copper salt transmits to a colorless flame. More characteristic, however, is the reaction of copper salts with ammonia. When such solutions are treated with concentrated ammonia water, the blue cupric oxide is precipitated. If, on the other hand, a copper sulphate is treated with diluted ammonia, the blue precipitate redissolves, with the formation of azure-blue solution. This solution contains ammoniated copper sulphate, having the following formula,

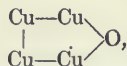


and is quite characteristic. It must be borne in mind, however, that nickel salts, when treated with ammonia, give a similar azure-blue solution. If metallic zinc or iron is brought in contact with a copper salt, an electrolytic action sets in, with the deposition of metallic copper on the other metal. This can be demonstrated by dipping into a copper solution a steel spatula, which, on removal, will be found plated with copper. As mentioned on p. 279, this test is a simple way for detecting the presence of copper in vegetable extracts.

Four oxides of copper are known— Cu_2O , Cu_2O , CuO , and CuO_2 .

Cu_2O (cuprous oxide) is a brick-red precipitate obtained by treating copper sulphate with an alkali in the presence of a reducing agent. It is best demonstrated in testing substances containing glucose (example, diabetic urine) with Fehling's solution. (See p. 695.)

Cupric oxide, CuO , occurs in the form of black granules or powder; Cu_2O , copper suboxide, graphically,



is an olive-green powder, and very rapidly oxidizes to the cuprous oxide, while copper superoxide, $\text{CuO}_2\cdot\text{H}_2\text{O}$, graphically,



is a yellow-brown powder.

But one salt of copper is official—

CUPRI SULPHAS. Copper Sulphate.



It should contain not less than 99.5 per cent. of pure Copper Sulphate [$\text{SO}_4\cdot\text{O}_2\text{Cu} + 5\text{H}_2\text{O}$].

Large, transparent, deep blue, triclinic crystals; odorless; of a nauseous, metallic taste; slowly efflorescent in dry air.

Soluble in about 2.2 parts of water, 400 parts of alcohol, and 3.5 parts of glycerin at 25° C. (77° F.) and in 0.5 part of boiling water.

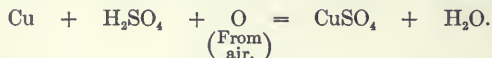
When heated to 30° C. (86° F.) the salt loses 2 of its 5 molecules of water (14.43 per cent.), and is converted into a pale blue, amorphous powder. Two more molecules of water are lost at 100° C. (212° F.), while the fifth is retained until 200° C. (392° F.) is reached, when a white, anhydrous powder remains (63.9 per cent. of the original weight). At a still higher temperature, sulphur dioxide and oxygen are given off, and a residue of black cupric oxide is left.

The aqueous solution (1 in 20) has a blue color and shows an acid reaction with blue litmus-paper.

Tests for Identity.—See Copper and Sulphates in Part V.

Impurities.—Excess of iron or of aluminum and heavy metals. Details in Part V.

Remarks.—This substance is known as *blue vitriol*, as already mentioned on p. 419. It is made by treating copper with sulphuric acid, and according as the acid is diluted or concentrated the reaction differs. When copper is treated with diluted sulphuric acid, the equation runs as follows:



while if the metal is treated with hot concentrated sulphuric acid, the equation is as given below:



It is interesting to note that in neither case is hydrogen produced, as usually occurs when a metal is treated with sulphuric acid.

Large amounts of crude blue-stone are made by roasting various ores and treating the residue with sulphuric acid.

Of the tests given above, its efflorescence and exsiccation are matter of interest; the fifth molecule of water of crystallization, held until the salt is heated to 200°C ., being an illustration of water of constitution (p. 147).

Copper sulphate, when given in small doses, acts as an astringent or tonic. If given in large doses, it acts as an emetic, and was formerly considerably used for this purpose. Its action, however, is too irritating to recommend its use in these days. Applied externally in the form of a moistened crystal it is caustic, while a dilute solution is used as an astringent injection in gonorrhea.

Dose.—Astringent, 10 milligrammes ($\frac{1}{4}$ grain); emetic, 250 milligrammes (4 grains).

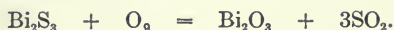
Cupric acetate, which was recognized in the pharmacopœia of 1880, is called *verdigris*, and was formerly in considerable use as an application for ring-worms and other skin troubles.

BISMUTH

Symbol, Bi. Atomic weight, approximately 207.

Bismuth is found in nature; it also occurs as sulphide in the ore, *bismuth glance*. The word bismuth is derived from the German *Wismuth*, the origin of this term being supposedly from the two German words *Weise matte*, beautiful meadow. This, however, has been pronounced by Huseman to be merely a pleasant fable, his researches proving that the first mention of the metal in the Bergbuchlein of 1518 was under the name of *Wyssmut*.

The extraction of the metal from the sulphide is accomplished by roasting in a reverberatory furnace, thus forming bismuth oxide, as shown in the following equation:



On heating bismuth oxide with coal, carbon dioxide is liberated, and metallic bismuth remains,



Bismuth is a crystalline, brittle, pulverizable, brilliant metal of silvery color, with, however, a slight reddish tint. In many of its qualities it resembles antimony.

The chief use of the metal is for the formation of fusible alloys, it possessing the characteristic property of blending with certain metals to form an alloy, melting at a temperature much below the melting-point of any of the metals when heated alone. Thus, two parts of bismuth, one part of lead, and one part of tin, the melting-point of the metals being respectively 270°C ., 330°C ., and 235°C ., melts at 94°C . (Rose's fusible metal), while Wood's metal, consisting of four parts of bismuth, one part of cadmium, one part of tin, and two parts of lead, melts at 60.5°C . The chief use of such fusible metals is for automatic fire-sprinklers, these consisting of water-pipes passing along the ceiling of a room, punctured with holes which are plugged with the fusible metal. If a fire breaks out in the apartment, as soon as the temperature reaches 65° , the metallic plugs melt and streams of water are thrown into the room. Pieces of these metals are also used as safety fuses in electric wiring.

Tests.—Bismuth, like so many metals of this group, gives a black sulphide when treated with hydrogen sulphide or ammonium sulphide. A more characteristic test is the fact that acid solutions of bismuth salts, when poured into water, produce a white precipitate of a "sub"-salt. Thus, bismuth nitrate solution, when poured into water, forms bismuth subnitrate, which precipitates out in the form of a white powder.

Three oxides of bismuth are known:

BiO or (Bi_2O_2) , which is a black substance, the graphic formula of which is



Bi_2O_3 , bismuthous oxide, a yellow powder; and Bi_2O_5 , which is red in color.

The bismuthous oxide is the one ordinarily found.

The following compounds of bismuth are official:

Bismuth citrate ($\text{BiC}_6\text{H}_5\text{O}_7$) should yield 58 to 60 per cent. absolute bismuth oxide.

Bismuth and ammonium citrate should yield at least 48 per cent. absolute bismuth oxide.

Bismuth subcarbonate—approximately $(\text{BiO})_2\text{CO}_3$ —should yield at least 90 per cent. absolute bismuth oxide.

Bismuth subgallate—approximately $\text{BiC}_7\text{H}_7\text{O}_7$ —should yield at least 52 to 57 per cent. absolute bismuth oxide.

Bismuth subnitrate—approximately BiONO_3 —should yield at least 80 per cent. absolute bismuth oxide.

Bismuth subsalicylate—approximately $\text{Bi}_2\text{O}_3(\text{C}_7\text{H}_5\text{O}_3)_3$ —should yield at least 62 to 64 per cent. absolute bismuth oxide.

In describing the official compounds of bismuth, the subnitrate is considered first, since from it almost all the other official bismuth preparations are made.

BISMUTHI SUBNITRAS. Bismuth Subnitrate.

Bismuth Subnitrate should yield not less than 80 per cent. of pure bismuth oxide.

A white powder, of somewhat varying chemical composition, odorless and almost tasteless, and permanent in the air.

Almost insoluble in water and insoluble in alcohol, but readily soluble in nitric or hydrochloric acid.

When heated to 120°C . (248°F .) for twelve hours the salt loses not over 3 per cent. of moisture; when subsequently heated to redness it evolves nitrous vapors, leaving not less than 80 per cent. of its weight of a yellow residue, which is soluble in nitric or hydrochloric acid and blackened by hydrogen sulphide.

When brought in contact with moistened blue litmus-paper the salt shows a slightly acid reaction.

On adding 3 Gm. of the salt to 3 Cc. of warm nitric acid no effervescence should occur (absence of *carbonate*), and no residue should be left (absence of *insoluble foreign salts*).

If this solution be poured into 100 Cc. of water, a white precipitate is produced.

Tests for Identity.—See above.

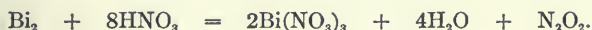
Impurities.—Excess chlorides, lead, copper, sulphate, silver, arsenic, and ammonia. Details in Part V. Alkalies and alkaline earths, see under bismuth subcarbonate. Carbonates, insoluble foreign salts. See above.

Assay.—Details in Part V.

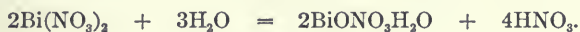
Remarks.—An ancient synonym for this preparation was *magisterium bismuthi*, or the master of bismuth, it being considered even in the days of the alchemists as the most important preparation of that metal. The present pharmacopœia gives no symbolic formula for bismuth subnitrate, as its composition varies. In the pharmacopœia of 1880 it was said to be $\text{BiONO}_3 \cdot \text{H}_2\text{O}$. The formula BiONO_3 can be resolved into a combination of Bi_2O_3 and bismuth nitrate,

$\text{Bi}(\text{NO}_3)_3$, viz., $\frac{\text{Bi}_2\text{O}_3}{\text{Bi}} (\text{NO}_3)_3$
 $\frac{\text{Bi}_2\text{O}_3(\text{NO}_3)_3}{\text{Bi}_2\text{O}_3(\text{NO}_3)_3}$, or 3BiONO_3 . The formula $\text{BiONO}_3 \cdot \text{H}_2\text{O}$ can also be considered as a combination of the hydroxide and the nitrate, viz., $\text{Bi} \begin{smallmatrix} \text{OH} \\ \text{OH} \\ \text{NO}_3 \end{smallmatrix}$. It is, therefore, apparent that the bismuth subnitrate is a mixture of the oxide and nitrate, even as ferric subsulphate was a combination of iron sulphate and iron oxide.

The bismuth subnitrate is prepared from bismuth nitrate, made by treatment of the metal with nitric acid, as is shown in the following equation:



This bismuth nitrate solution invariably contains arsenic, nearly all bismuth ores being contaminated with that metal, and in order to remove the arsenic, an elaborate process is necessary. The solution of nitrate is poured into sodium carbonate solution, when we obtain insoluble bismuth subcarbonate, while the arsenic present goes into solution in the form of sodium arsenate. The precipitate of bismuth subcarbonate is removed from the liquid by filtration, and then dissolved in nitric acid, when the nitrate is again formed. This is treated with a small quantity of water and allowed to stand twenty-four hours, when the remaining arsenic separates out in the form of crystalline bismuth subarsenate. The filtered bismuth nitrate solution is diluted with an equal quantity of distilled water, and to the mixture water of ammonia is slowly added with constant stirring. The pure bismuth subnitrate separates out, and the precipitate is collected on the filter, washed, and dried. In the final separation of the subnitrate the real action is due to the combination of bismuth nitrate with water, as is shown by the following equation:



The function of ammonia added is to neutralize the nitric acid separated in the reaction.

At present the salt is made by pouring a solution of chemically pure bismuth nitrate into water.

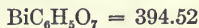
The pharmacopœia describes bismuth subnitrate as a heavy white powder. A special form of bismuth subnitrate, much esteemed in certain sections of the country, is a French subnitrate which occurs in the form of cones weighing 1.5 grammes, each cone being considered a medium adult dose. These cones are prepared by the process of trochiscation. (See p. 117.)

Bismuth subnitrate is used as an astringent and tonic, although if given in large doses it acts as a sedative, and is somewhat toxic.

Its action in diarrhea is largely mechanical, it forming a clammy covering for the inflamed mucous membrane, even as does prepared chalk.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

BISMUTH CITRAS. Bismuth Citrate.



Bismuth Citrate should yield not less than 58 per cent., nor more than 60 per cent., of pure bismuth oxide.

A white, amorphous or microcrystalline powder, odorless and tasteless, and permanent in the air.

Insoluble in water or alcohol, but soluble in ammonia water and in solutions of alkali citrates.

When strongly heated the salt chars, and, on ignition, leaves a more or less blackened residue, having a yellow surface and soluble in warm nitric acid; this solution, when dropped into a large excess of water, occasions a white turbidity.

Recipe and details of manufacture in Part VII.

Tests for Identity.—See Bismuth and Citrates in Part V.

Impurities.—Excess of nitrate, arsenic, lead, copper, silver, chlorides, and sulphates. Details in Part V.

Assay.—Details in Part V.

Remarks.—This official is made by heating bismuth subnitrate with citric acid and boiling for fifteen minutes, or until the test prescribed by the pharmacopœia shows that all the subnitrate has been converted into citrate. This process is an interesting illustration of how a chemical action not usually occurring can sometimes be produced. Ordinarily, the nitric acid combination of bismuth is much stronger than the combination of bismuth with citric acid. But if citric acid is boiled with bismuth subnitrate, it eventually drives off the stronger nitric acid by reason of the fact that the nitric acid is volatile, while the non-volatile citric acid remains in the mixture.

Bismuth subnitrate, as mentioned above, is insoluble in ammonia water, while the bismuth citrate is quite soluble therein, and in the official process the boiling is continued until the precipitate is found completely soluble in ammonia water.

Bismuth citrate is an example of a salt made by the union of a trivalent metal (Bi^{III}) with a tribasic acid, $\text{H}_3\text{C}_6\text{H}_5\text{O}_7$. One atom of Bi^{III} can replace the three atoms of hydrogen in the citric acid, $\text{H}_3\text{C}_6\text{H}_5\text{O}_7$, and thus we get bismuth citrate, $\text{BiC}_6\text{H}_5\text{O}_7$.

Bismuth citrate is a white powder, insoluble in water or alcohol, but soluble in ammonia water with the formation of the soluble bismuth and ammonium citrate. Bismuth citrate is rarely used in medicine, and about the only reason why it is given official recognition is because it is used in the manufacture of bismuth and ammonium citrate.

Dose.—125 milligrammes (2 grains).

BISMUTHI ET AMMONII CITRAS. Bismuth and Ammonium Citrate.

Bismuth and Ammonium Citrate should yield not less than 48 per cent. of pure bismuth oxide.

Shining pearly or translucent scales, odorless, having a metallic taste, and becoming opaque with loss of ammonia on exposure to the air.

Very soluble in water and sparingly soluble in alcohol.

When strongly heated the salt fuses, and finally leaves a blackened residue, having a yellow surface and soluble in warm nitric acid. The acid solution, when dropped into a large excess of water, occasions a white turbidity.

The aqueous solution of the salt is neutral or faintly alkaline to litmus-paper.

Recipe and details of manufacture in Part VII.

Tests for Identity.—See Bismuth, Ammonium, and Citrates in Part V.

Impurities.—Nitrate, excess chlorides, lead, copper, sulphates, silver, arsenic. Details in Part V. Alkalies and alkaline earths. Details under Bismuth Subcarbonate.

Assay.—Details in Part V.

Remarks.—This preparation is made by dissolving bismuth citrate in ammonia water and evaporating the filtered solution on the water-bath to syrupy consistence, and scaling the same by spreading it on plates of glass.

Bismuth and ammonium citrate is in the form of small pearly scales, becoming opaque on exposure to air. It is very soluble in water and but sparingly soluble in alcohol.

In order to secure the bismuth and ammonium citrate in the form of translucent scales it is necessary to spread it over thinly on glass, and it might be worth noting that the scales of bismuth and ammonium citrate are not as handsome as are the iron scale preparations—ferric citrate, for instance. If the solution is spread thickly on glass, white, opaque scales are formed, and, as has already been noted in the official description, often the pearly scales become opaque on exposure to air. A similar statement can also be made in regard to the solution of bismuth and ammonium citrate. This forms a clear, transparent liquid when freshly prepared, but on standing it becomes cloudy, due to gradual loss of ammonia, and, as the ammonia separates, insoluble bismuth citrate precipitates out. This cloudy solution can be cleared by the addition of a few drops of ammonia water.

Bismuth citrate is used as an astringent and tonic. It will be noted that the dose is much smaller than that of bismuth subnitrate. This is due to the fact that the bismuth and ammonium citrate is a soluble product, and it must be borne in mind that when bismuth compounds are soluble, they are by no means harmless. In fact, many therapeutists deprecate the administration of soluble bismuth compounds by reason of their possible toxic action. Bismuth and ammonium citrate was the basis of the elixir of bismuth and the elixir of pepsin and bismuth of the previous edition of the National Formulary. The recipe for the latter preparation was unsatisfactory, however, because of the incompatibility of bismuth and ammonium citrate combined with pepsin. Pepsin is held in solution by acid and precipitated by alkalies. On the other hand, bismuth and ammonium citrate is held in solution by alkalies and precipitated by acids. Therefore, if we add enough alkali to hold the bismuth citrate in solution, pepsin will be precipitated, while if we add acid to dissolve the pepsin, bismuth citrate will eventually precipitate. The present edition of the National Formulary lessens the possibility of incompatibility by using instead of the official salt, a glycerite of

bismuth which is a solution of bismuth and sodium tartrate, which double salt is soluble in acids and alkalis.

Dose.—125 milligrammes (2 grains).

BISMUTHI SUBCARBONAS. Bismuth Subcarbonate.

Bismuth Subcarbonate should yield not less than 90 per cent. of pure bismuth oxide.

A white or pale yellowish-white powder, of somewhat varying chemical composition, odorless and tasteless, and permanent in the air.

Insoluble in water or alcohol, but completely soluble in nitric or hydrochloric acid, with copious effervescence.

When heated to redness the salt loses water and carbon dioxide, and should leave not less than 90 per cent. of a yellow residue, which is soluble in nitric or hydrochloric acid and blackened by hydrogen sulphide T.S.

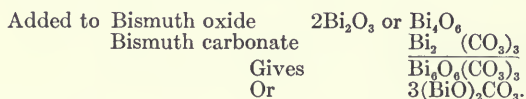
If 1 Gm. of the salt be boiled with 10 Cc. of a mixture of equal parts of acetic acid and water, the solution cooled and filtered, and the filtrate freed from bismuth by hydrogen sulphide, boiled, and again filtered, the last filtrate should leave no residue on evaporation (absence of *alkalies* and *alkali earths*).

Tests for Identity.—See Bismuth and Carbonates in Part V.

Impurities.—Chlorides, lead, copper, sulphate, silver, excess of subnitrate, arsenic. Details in Part V. Alkalies and alkali earths. See above.

Assay.—Details in Part V.

Remarks.—No formula for bismuth subcarbonate is given in the present pharmacopœia, but the standard of 1880 declared it to be $(\text{BiO})_2\text{CO}_3$. This formula may be resolved into bismuth oxide and bismuth carbonate, as is here shown:



Bismuth subcarbonate is prepared by dissolving metallic bismuth in nitric acid, and with careful handling the resulting bismuth nitrate solution is freed from the arsenic by diluting with a little water and allowing the mixture to stand in order to permit the subarsenate to separate out. This is filtered, and to the filtered liquid an equal quantity of water is added, and then a small amount of ammonia water, when the subnitrate precipitates. The bismuth subnitrate is redissolved in nitric acid, the filtered nitrate solution is then poured into a solution of sodium carbonate, and the precipitate of bismuth subcarbonate collected on the filter, washed, and dried.

Bismuth subcarbonate is used as an astringent and tonic, and in use resembles bismuth subnitrate.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

BISMUTHI SUBGALLAS. Bismuth Subgallate.

Bismuth Subgallate should yield not less than 52 nor more than 77 per cent., of pure bismuth oxide.

An amorphous, bright yellow powder, somewhat variable in chemical composition, without odor or taste, and permanent in the air.

Insoluble in water, alcohol, and ether; readily soluble with decomposition in hydrochloric, nitric, and sulphuric acids, if these be heated; insoluble in very dilute mineral acids; readily soluble in solutions of the alkali hydroxides, forming a clear yellow-colored solution, which rapidly changes to a deep red.

When heated to 120° C. (248° F.) the salt loses from 5 to 7 per cent. of water, and on subsequent heating to redness it at first chars, finally leaving a yellow residue, which is soluble in hydrochloric and nitric acids and is blackened by ammonium sulphide T.S.

Tests for Identity.—See Bismuth and Gallates in Part V.

Impurities.—Free gallic acid, excess of nitrates, chlorides, lead, copper, sulphate, silver, and arsenic. Details in Part V.

Assay.—Details in Part V.

Remarks.—This new official is prepared by treating an acetic acid solution of bismuth nitrate with gallic acid.

It is popularly known as *dermatol*, and is used as an astringent, both externally and internally.

Dose.—250 milligrammes (4 grains).

BISMUTHI SUBSALICYLAS. Bismuth Subsaliolate.

Bismuth Subsaliolate should yield not less than 62 nor more than 64 per cent. of pure bismuth oxide.

A white or nearly white, amorphous or crystalline powder, odorless, tasteless, and permanent in the air.

Almost insoluble in cold water; upon prolonged boiling with water a portion of the salicylic acid passes into solution, with the formation of a more basic bismuth salicylate. It is partly soluble with decomposition in hydrochloric and nitric acids, a white, flocculent precipitate of salicylic acid separating.

When heated to 120° C. (248° F.) Bismuth Subsaliolate should not lose more than 1 per cent. of water, and on subsequently heating to redness it at first chars, finally leaving a yellow residue, which is soluble in hydrochloric or nitric acid and is blackened by ammonium sulphide T.S.

Tests for Identity.—See Bismuth and Salicylates in Part V.

Impurities.—Free salicylic acid, excess of nitrates, chlorides, lead, copper, sulphate, silver, and arsenic. Details in Part V.

Assay.—Details in Part V.

Remarks.—Bismuth subsaliolate is made by triturating and warming an aqueous solution of salicylic acid with anhydrous bismuth oxide, obtained by boiling bismuth hydroxide which has been prepared by precipitating a nitric acid solution of bismuth subnitrate with potassium hydroxide or ammonium hydroxide. It is used as an intestinal antiseptic.

Dose.—250 milligrammes (4 grains).

CHAPTER XXXIV

LEAD, MERCURY, AND SILVER

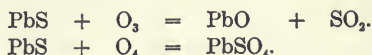
LEAD

Symbol, Pb. Atomic weight, 205.35

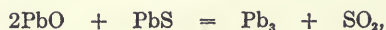
LEAD occurs in the form of sulphide in the ore *galena*. By way of passing it is worth noting that some galena ore contains considerable silver. (See p. 621.) Among the other ores of lead are various forms of the carbonate and the sulphate.

Metallic lead was used in classic times, the Latin name *plumbum* being the same applied to it by classic writers, its original meaning being "heavy."

Lead is prepared from the sulphide by roasting in a reverberatory furnace, thus converting the sulphide, under the influence of atmospheric oxygen, into lead oxide and lead sulphate, shown in the following equations:



The resulting lead oxide combines with a new portion of lead sulphide with the formation of the metal and the separation of SO_2 , by the following equation:



while the lead sulphate reacts with more lead sulphide in the manner shown in the following equation:



Lead is a heavy, bluish metal, freely malleable, and somewhat ductile. Its pliability has given rise to its extended use in plumbing, the name of the craft being derived from the word *plumbum*, lead, since leaden pipes are largely used for the transport of water. It should be said that lead water-pipes are scarcely harmless, traces of lead hydroxide dissolving, and water so contaminated produces a dangerous ailment called lead colic. With hard water, *i. e.*, water containing lime and magnesia dissolved by aid of carbon dioxide, there is less danger, as carbonic acid acts on fresh lead, forming lead carbonate, which coats the interior of the pipe and prevents any of the lead dissolving in the water. Lead is used in the manufacture of *solder*, which is a variety of the fusible metal made by combining equal quantities of tin and lead. This mixture melts at 186°C. , and is used for fastening pieces of tin or other metal together.

The *tests* for lead are as follows: Hydrochloric acid produces a white precipitate of lead chloride, being one of the three chlorides thus precipitated, the other two metals precipitated in the form of chloride being silver and mercurous compounds. Lead gives with hydrogen sulphide and ammonium sulphide a black sulphide; with sulphuric acid it precipitates white lead sulphate, and with carbonates, white lead carbonate is precipitated out of the solution. Quite characteristic is the yellow precipitate of lead chromate produced when a solution of lead salt is treated with a solution of potassium chromate.

Five oxides of lead are known, Pb_2O , a black velvety powder, expressed by the graphic formula,



Pb_2O_3 , which may be considered as a mixture of PbO and PbO_2 , the composition of which can best be explained by the following graphic formula, $\text{Pb} \begin{array}{c} \text{O} \\ \text{=} \\ \text{O} \end{array} \text{Pb}$, that is, a combination of lead with valence iv,

with lead with valence II; PbO_2 , which is called *puce-colored lead oxide*, in which lead shows valence IV; Pb_3O_4 , or *red lead*, which can be considered as a combination of Pb_2O_3 and PbO , or else as a lead plumbate, Pb_2PbO_4 ; that is, the lead salt of orthoplumbic acid, $\text{Pb}(\text{OH})_4$ or H_4PbO_4 . Red lead is made by sprinkling hot litharge with water and carefully drying. It is of value as a pigment.

The official preparations of lead are:

Lead acetate. At least 99.5 per cent. absolute $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3\text{H}_2\text{O}$.

Solution of lead subacetate. At least 25 per cent. absolute $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Cerate of lead subacetate contains 20 per cent. solution of lead subacetate.

Diluted solution of lead subacetate contains about 1 per cent. lead subacetate.

Lead iodide. At least 99 per cent. absolute PbI_2 .

Lead nitrate. At least 99.5 per cent. absolute $\text{Pb}(\text{NO}_3)_2$.

Lead oxide. At least 96 per cent. absolute PbO .

Lead plaster. A lead oleopalmitate.

Diachylon ointment contains 50 per cent. lead plaster.

PLUMBI OXIDUM. Lead Oxide.

$\text{PbO} = 221.23$

It should contain not less than 96 per cent. of pure Lead Oxide and should be kept in well-closed vessels.

A heavy, yellowish or reddish-yellow powder, or minute scales, without odor or taste. On exposure to the air it slowly absorbs moisture and carbon dioxide.

Almost insoluble in water, to which it, however, imparts a faintly alkaline reaction; insoluble in alcohol, but soluble in acetic or diluted nitric acid and in warm solutions of the fixed alkali hydroxides.

When heated the Oxide assumes a brownish-red color, becoming yellow again on cooling; it fuses at a red heat. When heated in contact with charcoal it is reduced to metallic lead.

Lead Oxide should be soluble in diluted nitric acid, with but little effervescence (limit of *carbonate*) and without the development of the odor of nitrous acid (absence of *lead*), leaving not more than a trifling residue (limit of *silicates*, *barium sulphate*, etc.).

The solution in diluted nitric acid, which should be colorless, when nearly neutralized by ammonia water yields with hydrogen sulphide T.S. a black precipitate, with potassium iodide T.S. a yellow one, and with diluted sulphuric acid a white precipitate, the latter two being soluble in a strong solution of sodium hydroxide.

If from the solution in diluted nitric acid the lead be precipitated by sulphuric acid, the filtrate, after the addition of an excess of ammonia water, should not assume more than a slight bluish tint (limit of *copper*), nor yield more than traces of a reddish-brown precipitate (limit of *iron*).

If 5 Gm. of the Oxide contained in a small flask be shaken with 5 Cc. of water, then 20 Cc. of acetic acid added, and the mixture boiled for a few minutes and filtered, the insoluble residue, when well washed and dried, should not weigh more than 0.2 Gm. (absence of more than 4 per cent. of *insoluble impurities*).

If, to the mixed filtrate and washings obtained in the last test, hydrochloric acid be added until no further precipitate be produced, the remainder of the lead removed from the filtrate by hydrogen sulphide, and the liquid filtered, the second filtrate, upon evaporation to dryness, should not yield a residue weighing more than 0.050 Gm. (limit of *soluble impurities*).

When strongly heated in a porcelain crucible, the Oxide should not lose more than 4 per cent. of its weight (limit of *carbonate* and of *moisture*).

Tests for Identity.—See above.

Impurities.—Carbonates; metallic lead; excess silicates, barium sulphate, copper, iron, soluble impurities, carbonate, and moisture; more than 4 per cent. insoluble impurities. Details above.

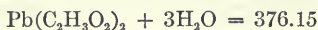
Remarks.—This oxide of lead is called *litharge*, a word used since ancient times, and derived from the two Greek words, *lithos*, a stone,

and *arguros*, silver. It is so called because the ancients discovered that when the ore, crude litharge, was fused, the pure litharge, freed from the mineral impurities, fused in the form of bright, shiny, silvery scales.

Manufacture.—The chief source of litharge is as a side-product in obtaining silver from galena. The ore (lead sulphide, p. 597) is calcined, thus driving off the sulphur, and the residue brought to a state of fusion, when the lead oxide separates out by crystallization from the fused mass of silver.

Litharge is but little used in medicine, although important as a constituent of two popular pharmaceuticals, solution of lead subacetate (U. S. P. VIII) and lead plaster, as made by process of U. S. P. 1890.

PLUMBI ACETAS. Lead Acetate.



It should contain not less than 99.5 per cent. of pure Lead Acetate $[(\text{CH}_3\text{COO})_2\text{Pb} + 3\text{H}_2\text{O}]$ and should be kept in well-stoppered bottles.

Colorless, shining, transparent, monoclinic prisms or plates, or heavy, white, crystalline masses, or granular crystals, having a faintly acetous odor and a sweetish, astringent, afterward metallic taste. Efflorescent and absorbing carbon dioxide on exposure to the air.

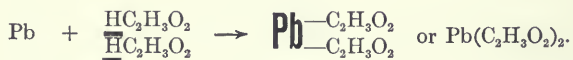
Soluble in 2 parts of water and in 30 parts of alcohol at 25° C. (77° F.); in 0.5 part of boiling water and in 1 part of boiling alcohol.

When heated to 40° C. (104° F.) the salt loses its water of crystallization (14.26 per cent.). When heated rapidly to 75° C. (167° F.) it fuses in its water of crystallization, and at a higher temperature is converted into a pulverulent basic salt which fuses at about 280° C. (536° F.), with continued loss of acetic acid, finally decomposing with the evolution of carbon dioxide and acetone, leaving a residue of finely divided metallic lead mixed with oxide and carbonate.

Tests for Identity.—See Lead and Acetates in Part V.

Impurities.—Excess of carbonate (solution with boiled water should be clear or only slightly opalescent). Iron, copper, zinc. Details in Part V. Salts of the alkalis, magnesium, calcium, etc. (hydrochloric acid solution on evaporation should leave no residue).

Remarks.—Lead acetate is incorrectly and dangerously called "*sugar of lead*." Lead being bivalent, it can replace two hydrogen atoms in an acid, and since acetic acid has but one replaceable hydrogen atom to the molecule, two molecules of acetic acid are needed to combine with one atom of lead, thus:



However, the acetate is usually made by treating the oxide litharge (*not the metal*) with acetic acid.

The solution is concentrated, and the lead acetate is allowed to crystallize. It is important to note that the average commercial lead acetate should not be dispensed without purification, as it contains appreciable quantities of both lead oxide and lead carbonate. The purification of this commercial lead acetate is a simple matter, however, the acetate dissolving in water and recrystallizing, while the impurities do not dissolve.

Lead acetate is used as an astringent and sedative, both internally

and externally. An overdose acts as an irritant poison, producing severe gastric disturbances. The antidote for lead-poisoning or in lead-colic is an alkaline sulphate, the one generally preferred being magnesium sulphate or Epsom salt. Externally, it is used either in an aqueous solution or in the form of suppositories, the latter being a highly esteemed remedy for piles, a favorite prescription of this kind being lead acetate with extract of opium and oil of theobroma. The preparation of the suppositories of lead and opium requires considerable skill.

The extract of opium should be rubbed up with enough water to make it into a paste thin enough to pour. The lead acetate should be reduced to the form of an impalpable powder, and added to the molten oil of theobroma, with constant stirring, the mixture is then poured into molds, after adding the thinned extract, just at that moment when the oil of theobroma is ready to solidify. Unless these precautions are followed (especially the stirring), there is danger of the heavy lead acetate settling at the bottom of the dish in which the oil of theobroma is melted, and thus the last suppository poured therefrom will contain a majority of the lead salt. Lead acetate solution is used as a wash for some skin troubles, such as ivy-poisoning and chilblains, but for this purpose a solution of lead subacetate is frequently preferred.

Dose.—65 milligrammes (1 grain).

LIQUOR PLUMBI SUBACETATIS. Solution of Lead Subacetate.

An aqueous liquid, which should contain in solution not less than 25 per cent. of Lead Subacetate [approximately $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{COO})_2 = 543.74$].

A clear, colorless liquid, odorless, having a sweetish, astringent taste and an alkaline reaction. On exposure to the air it absorbs carbon dioxide, which causes the formation of a white precipitate.

Specific gravity: about 1.235 at 25° C. (77° F.).

When Solution of Lead Subacetate is added to a solution of acacia, it produces a dense, white precipitate (distinction from *normal lead acetate*).

Recipe and details of manufacture in Part VII.

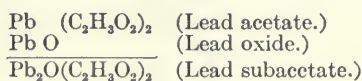
Tests for Identity.—Same as under Lead Acetate.

Impurities.—Same as under Lead Acetate.

Assay.—Details in Part V.

Remarks.—This preparation—*Goulard's extract*—contains lead subacetate, which, like all "sub-" salts (see Ferric Subsulphate, p. 561), is a combination of a salt of a metal with the oxide of the same metal. Remember it is not a mere mechanical mixture, but a combination like that existing between the sulphates of aluminum and potassium in official alum.

The solution is made by boiling together water, lead acetate, and lead oxide; full details of manufacture and equation, showing chemical action, being given on p. 1131. The pharmacopœia says that the solution contains approximately $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$, which we find corresponds to the sum resulting from the addition of one molecule of lead acetate to one molecule of lead oxide, viz.:



That a chemical change is produced is shown by the fact that the insoluble lead oxide is made to dissolve with the formation of a soluble oxyacetate.

It will be noted that the lead subacetate solution is unstable, especially unless kept tightly corked, the carbon dioxide of the air acting on the solution, with the formation of lead carbonate which precipitates.

Solution of lead subacetate is used almost entirely externally; for this purpose being employed as a sedative and astringent wash in minor skin troubles, such as bruises, frost-bite, and the like. For many purposes of this kind a diluted solution is preferable, and that is provided by the pharmacopœia.

LIQUOR PLUMBI SUBACETATIS DILUTUS. Diluted Solution of Lead Subacetate.

Lead-water.—This is made by diluting four parts of the strong solution of lead subacetate, given above, with 96 parts of water, which has been previously boiled in order to expel the carbon dioxide it might contain. (See p. 385.) This solution is usually dispensed in a slightly opalescent condition, first because, on standing, it will gradually become opalescent anyway, and, secondly, to afford a differentiation between the so-called “lead-water” and “lime-water,” the latter product being dispensed in a crystal-clear form.

Lead-water is used as a soothing lotion, and is frequently dispensed in combination with a small quantity of tincture of opium. This mixture, while quite popular, is incompatible, for, as soon as the tincture of opium is added to the lead-water, there is formed a flocculent precipitate (lead meconate). The prescription is a usual one, however, and pharmacists always dispense the same, containing the precipitate, placing a “Shake Well!” label on the bottle.

CERATUM PLUMBI SUBACETATIS. Cerate of Lead Subacetate.

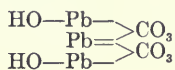
This is made by incorporating solution of lead subacetate with an ointment base consisting of wool-fat, paraffin, white petrolatum, and camphor. (See p. 341.) It, like the solution from which it is made, was first introduced by the chemist Goulard, hence it is called *Goulard's cerate*. That formerly official, made with camphor cerate, which contained lard, soon assumed a yellow color, and in a very short time became too rancid to use. The present official, made with wool-fat and petrolatum fats, does not decompose so easily.

Goulard's cerate is used for mild skin troubles, and is particularly recommended for cases of chilblain.

PLUMBI CARBONAS (U. S. P. 1890). Lead Carbonate.

Lead carbonate, or *white lead*, like the carbonates of zinc and of magnesium, is a combination of the carbonate and the hydroxide; the formula given the compound in the pharmacopœia of 1890 having been $(\text{PbCO}_3)_2\text{Pb}(\text{OH})_2$.

It must be borne in mind that such a formula as the one just given does not represent a mechanical mixture of lead carbonate and lead hydroxide, but a combination far more intimate, which may be best explained by the following graphic formula:



Lead carbonate can be made in small quantities at the prescription counter by combination of molecular quantities of lead nitrate and sodium carbonate, but that used for paint is produced by other means. The oldest method and one still largely used, because of the superiority of the product, as a paint, is that made by the so-called *Dutch process*. By this process sheets of lead are coiled in earthenware pots, moistened with acetic acid. A long series of these pots, carefully covered to prevent the admission of dirt, and yet loose enough to permit the admission of gases, are then placed in rows, are covered with decaying manure or other animal matter, and allowed to stand so covered for five to six weeks. The decaying animal matter produces a large quantity of carbon dioxide, which is gradually absorbed by the lead acetate first produced in the pots by the action of acetic acid on the sheet lead. Experience tells how long the maceration should continue, and when a sufficient length of time has elapsed, the covering of manure is removed, and the contents of the pots are found to consist of powdered white lead. Besides this Dutch process, large quantities of white lead are now made for paints by passing carbon dioxide through a solution of lead subacetate.

Lead carbonate is indicated in some cases of skin trouble and is then usually applied in the form of ointment (*unguentum plumbi carbonatis*, U. S. P. 1890). This consists of 10 Gm. of lead carbonate in a very finely powdered form, rubbed up with 90 Gm. of benzoinated lard.

A considerable amount of white lead was formerly used as a cosmetic (face powder). Its use for this purpose is dangerous, because of the absorption of the lead through the pores of the skin, which causes lead colic.

Rubbed to a fine paste with linseed oil, white lead constitutes one of the most popular white paints, although such paints should not be used in lavatories or other apartments where there is a possibility of the liberation of hydrogen sulphide gas, which will change the white lead carbonate into the black lead sulphide.

PLUMBI IODIDUM. Lead Iodide.

$$\text{PbI}_2 = 457.15$$

It should contain not less than 99 per cent. of pure Lead Iodide and should be kept in well-stoppered bottles, protected from light.

A heavy, bright yellow powder, without odor or taste; permanent in the air.

Soluble in about 1300 parts of water at 25° C. (77° F.) and in about 200 parts of boiling water, separating from the latter solution on cooling in brilliant golden-yellow crystalline laminae; very slightly soluble in alcohol, but soluble, without color, in solutions of the fixed alkalies, in concentrated solutions of the alkali acetates, of potassium iodide, and of sodium thiosulphate, and in a hot solution of ammonium chloride.

When moderately heated the salt fuses to a thick, reddish-brown liquid, which congeals, on cooling, to a yellow, crystalline mass. At a higher temperature it is decomposed, with the evolution of violet vapors of iodine, leaving a lemon-yellow residue of lead oxyiodide.

If 1 Gm. of the salt be triturated with 2 Gm. of ammonium chloride and 2 Cc. of water, a nearly white mixture will result. If this be transferred to a test-tube, and heated in a water-bath for a few minutes, a clear and almost colorless solution should be formed (absence of *chromate* and *other insoluble foreign salts*). On cooling this solution a solid mass of nearly colorless, fine, silky crystals will be produced, and on adding water or diluted sulphuric acid to this mass, yellow Lead Iodide will separate.

Tests for Identity.—See above.

Impurities.—Chromate and insoluble foreign salts, see above. Excess of nitrates, acetates. Details in Part V. Soluble foreign salts (aqueous filtrate, on evaporation, should yield no residue).

Remarks.—Lead iodide, PbI_2 , shows lead with valence II, the linkage being,



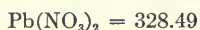
This chemical is made by treating a solution of lead nitrate with potassium iodide, as shown in the following equation,



and collecting and washing the precipitated lead iodide.

Lead iodide is rarely used internally. When it is, it acts as an alternative, and is given in doses of one to three grains. Its chief value is for external use in tumors and indolent swellings, in which case it is usually employed in the form of the *unguentum plumbi iodidum* (U. S. P. 1890), which is made by rubbing up ten parts of very finely powdered lead iodide with 80 parts of benzoinated lard.

PLUMBI NITRAS. Lead Nitrate.



It should contain not less than 99.5 per cent. of pure Lead Nitrate $[(\text{NO}_2)_2\text{O}]_2\text{Pb}$ and should be kept in well-closed vessels.

Colorless, transparent, octahedral crystals when obtained by the spontaneous evaporation of cold solutions, or white, nearly opaque crystals when formed by the cooling of hot solutions; without odor and having a sweetish, astringent, afterward metallic taste; permanent in the air.

Soluble in 1.85 parts of water at 25° C. (77° F.) and in 0.75 part of boiling water; almost insoluble in alcohol.

When strongly heated the salt decrepitates, emits nitrous vapors, and finally leaves a residue of lead oxide.

Tests for Identity.—See Lead and Nitrates in Part V.

Impurities.—Copper, iron, zinc. Details in Part V. Salts of the alkalis, magnesium, calcium, etc. See Lead Acetate.

Remarks.—This chemical is made by treating lead oxide with nitric acid, as shown in the following equation:



The solution of lead is concentrated by the evaporation of the surplus water, and after filtering it is allowed to stand, when the lead nitrate separates in the form of small crystals.

The use of lead nitrate is for the preparation of the other salts of lead. Medicinally, it is used externally as a cooling wash, and enjoys some reputation as a disinfectant.

EMPLASTRUM PLUMBI. Lead Plaster.

For recipe, see Part VII.

Remarks.—The recipe for the manufacture of this pharmaceutical was changed in the last pharmacopœial revision; that of the pharmacopœia of 1890 being given in full details in Part VII. (p. 1132), where the chemistry of manufacture will be found. The process of the present pharmacopœia will also be found in Part VII., so here it need only be said that it is made by mixing together aqueous solutions of lead acetate and of Castile soap. This method of manufacture can be explained by the following diagram:



No attempt is here made to express the reaction by means of symbolic formula, since the complex chemistry of soap and of lead plaster can be best reserved for the appropriate chapters in organic chemistry (p. 739).

As the name indicates, the product is used as a plaster base.

UNGUENTUM DIACHYLON. Diachylon Ointment.

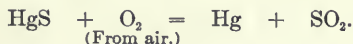
(See p. 346.) This ointment consists of melted lead plaster diluted with olive oil and oil of lavender. The ointment was formerly in considerable repute as a remedy for eczema.

MERCURY

Symbol, Hg. Atomic weight, 198.5

Mercury occurs chiefly as a sulphide, forming the valuable mineral *cinnabar*, and is occasionally found in the native state. Cinnabar has been known since the time of the Grecian philosophers, and the separation of the metal by heating of cinnabar was described by Dioscorides, while the metal was called hydrargyrum (water silver) by Pliny. It was first used in medicine by Paracelsus.

Metallurgy.—Mercury is obtained by roasting cinnabar in an appropriate retort, when mercury and sulphurous oxide are formed, as shown in the following equation:



The vapors of sulphurous oxide are permitted to escape, while the metallic mercury is held back in an appropriate condenser.

Mercury is a silvery white, mobile liquid, hence called *quicksilver*. It has a specific gravity of 13.59, and is the heaviest known liquid. It is, moreover, of interest as being the only metallic substance which is liquid at ordinary temperatures. It requires of cold 40° below zero (both Fahrenheit and Centigrade) to solidify it.

Mercury has the property of combining with almost all the metals (but iron), forming liquid alloys, which we call amalgams; some of which are definite chemical compounds (example, K₂Hg). The fact that iron does not combine with mercury, permits the storage of mercury in iron containers. Mercury boils at 360° C., but vaporizes below that temperature, and the vapor is poisonous. Hence trades demanding the use of a large amount of mercury, such as in making mirrors, are dangerous to the health of the workmen. As mentioned on p. 363, the atomic and molecular weight of mercury is the same, 200.

The tests for mercury are as follows: The black sulphide produced by the action of hydrogen sulphide or ammonium sulphide; a routine test, which is of very little value, except in the precipitation of mercury in the regular scheme of analytic chemistry. (See p. 984.) In this test both forms of mercury (the "ous" and "ic") separate in the form of black mercuric sulphide. There are quite a number of characteristic tests differentiating the "ous" compounds from the "ic" compounds. The Potassium iodide precipitates from the solution of mercurous salts the yellow mercurous iodide; while the same reagent with the mercuric salts gives a red mercuric iodide.

Hydrochloric acid precipitates from solutions of mercurous salts insoluble white mercurous chloride; while with "ic" salts no precipitate is formed by this reagent. Metallic copper or stannous chloride solution, when mixed with a mercurous compound, reduces it to a metallic mercury, which separates out in the form of a fine gray precipitate. Calcium hydroxide solution (lime-water) forms with mercurous compounds a black precipitate of mercurous oxide,— Hg_2O ,—and the liquid so prepared from 13 grains calomel and 3 fluidounces lime-water is called *black wash* (*aqua phagedænica nigra*, *lotio nigra*, N. F.). Mercuric salts with lime-water form a yellow precipitate of mercuric oxide, and liquid so prepared from 5 grains corrosive sublimate and 3 fluidounces lime-water is called *yellow wash* (*aqua phagedænica*, *lotio flava*, N. F.).

Two oxides of mercury are known— Hg_2O , mercurous oxide, and HgO , mercuric oxide.

Referring to the table of elements on p. 367, we find that mercury is both univalent and bivalent, and we, therefore, have two lines of mercury salts—the mercuric and mercurous.

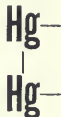
When speaking of copper on p. 590, attention was called to the fact that metal exhibited the valence I and II, and that such variation did not agree with the rule, laid down on p. 365, that variation in valence should be by twos. Such exception we found in the ferric and ferrous salts, the apparent valence in that case being II and III, and that exception was explained on p. 548 by the theory that ferric iron really has the valence IV, making iron, II and IV. In a somewhat similar way we explain the valence of copper and mercury. In the mercuric form the valence is undoubtedly II, and mercuric chloride can be expressed as shown in Formula A, given below.

In the mercurous form the valence is also supposed to be II, and all mercurous compounds are supposed to be made up of two atoms of mercury linked as in Formula B. Thus we suppose the formula of mercurous chloride (calomel) is as in Formula C,

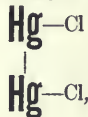
FORMULA A.



FORMULA B.



FORMULA C.



and the pharmacopœia of 1890 gave the formula of calomel as Hg_2Cl_2 , not HgCl , and likewise with all other mercurous salts.

The theory of the bivalency of mercury in mercurous compounds is not, however, unanimously accepted, some chemists considering mercury in the "ous" form as possessing valence I, and thus giving the mercurous bodies the simpler single formula—for example, calomel, HgCl . This view is the one recognized in the new pharmacopœia (U. S. P. VIII.), where all formulas (see Alum, p. 535, and Ferric Chloride, p. 548) are given their simplest form.

The official forms of mercury and its compounds are:

Mercury. At least 99.9 per cent. absolute Hg.

Mass of mercury contains 33 per cent. mercury.

Mercurial ointment contains 50 per cent. mercury.

Blue ointment contains 33 per cent. mercury.
 Mercurial plaster contains 30 per cent. mercury.
 Mercury with chalk contains 38 per cent. mercury.
 Ammoniated mercury— HgNH_2Cl —contains 78 to 80 per cent. mercury.
 Ointment of ammoniated mercury contains 10 per cent. ammoniated mercury.
 Corrosive mercuric chloride. At least 99.5 per cent. absolute HgCl_2 .
 Mild mercurous chloride. At least 99.5 per cent. absolute HgCl .
 Red mercuric iodide. At least 99.5 per cent. absolute HgI_2 .
 Yellow mercurous iodide. At least 99.5 per cent. absolute HgI .
 Red mercuric oxide. At least 99.5 per cent. absolute HgO .
 Ointment of red mercuric oxide contains 10 per cent. red mercuric iodide.
 Yellow mercuric oxide. At least 99.5 per cent. absolute HgO .
 Ointment of yellow mercuric oxide contains 10 per cent. yellow mercuric oxide.
 Oleate of mercury contains 25 per cent. yellow mercuric oxide.
 Solution of mercuric nitrate contains 60 per cent. mercuric nitrate [$\text{Hg}(\text{NO}_3)_2$].
 Ointment of mercuric nitrate contains 7 per cent. mercury combined as nitrate.

HYDRARGYRUM. Mercury.

Hg = 198.50

It should contain not less than 99.9 per cent. of pure metallic Mercury, and should be kept in strong, well-stoppered bottles.

A shining, silver-white metal, without odor or taste.

It is liquid at ordinary temperatures and easily divisible into spherical globules; but when cooled to -39.38°C . (-38.88°F .) it forms a ductile, malleable mass.

Specific gravity: 13.535 at 25°C . (77°F .)

Insoluble in the ordinary solvents, also in concentrated hydrochloric acid, and, at ordinary temperatures, in sulphuric acid; but it dissolves in the latter when boiled with it, and is readily and completely soluble in nitric acid.

At ordinary temperatures it volatilizes very slowly, more rapidly as the temperature increases, and at 357.25°C . (675.05°F .) it boils and is completely volatilized, yielding a colorless, very poisonous vapor, and no appreciable residue.

When globules of Mercury are dropped upon white paper, they should roll about freely, retaining their globular form, and leaving no streaks or traces.

It should be perfectly dry and present a bright surface even after agitation in contact with air.

On boiling 5 Gm. of Mercury with 5 Cc. of water and 4.5 Gm. of sodium thio-sulphate, in a test-tube, for about one minute, the Mercury should not lose its lustre, and should not acquire more than a slightly yellowish shade (absence of more than slight traces of foreign metals).

Tests for Identity.—See above.

Impurities.—Excess of foreign metals. See above.

Remarks.—Metallic mercury is one of the three metals recognized by the pharmacopœia as a metal, the other two being zinc and iron.

It will be noticed from the pharmacopœial description of the metal that one of the tests is that a globule of mercury rolled on paper should not leave a black mark. If it does leave a mark, it means that the mercury is contaminated with dirt, and this can be removed by straining through chamois. If there is only a small quantity of mercury to be so cleaned, it can be done by placing a plain filter in a funnel, and piercing the point of the filter with a pinhole, just large enough to permit the passage of the mercury in a very fine stream.

The globules of mercury rolling down over the paper toward the pinhole will leave most of the dirt against the paper.

It must be borne in mind, however, that this process will remove only such impurities as are insoluble, and for a complete purification of the metal its distillation is advisable. To perform this is, however,

a matter of difficulty, except on a large scale, and recourse is had to purification by treatment of diluted nitric acid, washing with water, drying, and expressing through chamois.

Mercury is always given medicinally in finely divided form, and of such preparations the following are official:

MASSA HYDRARGYRI. Mass of Mercury.

If a portion of the Mass be triturated in a mortar, with warm acetic acid, the filtrate should not become more than slightly opalescent on the addition of a few drops of hydrochloric acid (limit of *mercurous oxide*).

If another portion of the Mass be digested with warm, diluted hydrochloric acid and a little purified animal charcoal, the filtrate should not be affected by hydrogen sulphide T.S. or by stannous chloride T.S. (absence of *mercuric oxide*).

Recipe and details of manufacture see Part VII.

Impurities.—Excess of mercurous oxide; mercuric oxide, see above.

Remarks.—*Mass of mercury* is mercury extinguished (as already explained on p. 311) by honey of rose and glycerin, the excipient (a hardener in this case) being glycyrrhiza and althæa. For details of manufacture see p. 1102. Mass of mercury is used as a cholagogue.

Dose.—250 milligrammes (4 grains).

HYDRARGYRUM CUM CRETA. Mercury with Chalk.

<i>Recipe</i> .—Mercury, thirty-eight grammes.....	38 Gm.
Clarified Honey, ten grammes.....	10 Gm.
Prepared Chalk, fifty-seven grammes.....	57 Gm.
Water, a sufficient quantity,	

To make one hundred grammes. . 100 Gm.

Weigh the Mercury and Clarified Honey successively into a strong bottle of the capacity of one hundred cubic centimeters, and add two cubic centimeters of Water. Cork the bottle, and shake it for about half an hour at a time, until the aggregate time of shaking reaches ten hours, or until the globules of Mercury are no longer visible under a lens magnifying four diameters. The shaking may be more conveniently performed by mechanical means. Rub the Prepared Chalk with Water, in a mortar, to a thick, creamy paste, and, having added the contents of the bottle, washing the last portions in with a little Water, triturate the whole to a uniform mixture. Finally, dry the mixture, first between ample layers of bibulous paper, and afterward in a dish at the ordinary temperature, until it weighs one hundred grammes. Then reduce it to a uniform powder, without trituration, and keep it in well-stoppered bottles, protected from light.

A light gray, rather damp powder, free from grittiness, without odor, and having a slightly sweetish taste.

Impurities.—Mercurous and mercuric oxides. See Mass of Mercury.

Remarks.—*Mercury with chalk* is a finely divided form of mercury, containing 38 per cent. of metal extinguished by clarified honey, moist chalk being then added. Like all mercurial preparations made of the free metal, the extinguishing is the most tiresome, and in this special case can only be performed satisfactorily by machinery. In the finished product the mercury globules should not be seen with a glass magnifying four diameters.

This body is used as a cholagogue.

Dose.—250 milligrammes (4 grains).

UNGUENTUM HYDRARGYRI. Mercurial Ointment.*Recipe.*—See p. 346.**ASSAY OF MERCURIAL OINTMENT**

Weigh 10 Gm. of Mercurial Ointment in a tared dish, melt it, then remove it from the fire, and add 50 Cc. of warm petroleum benzin. Stir the mixture well, allow the Mercury to settle completely, and decant the petroleum benzin. Wash the residue with successive portions of 10 Cc. each of warm petroleum benzin until it is entirely free from fatty matter, carefully retain all of the separated Mercury in the dish, and allow all traces of the benzin to evaporate. Add to the residue 10 Cc. of diluted hydrochloric acid, heat it gently, and stir with a glass rod until the Mercury collects in a globule. Pour off the acid, warm the Mercury with a little distilled water, dry the globule on bibulous paper, and weigh. The Mercury should weigh not less than 4.9 Gm.

Ointment of mercury contains 50 per cent. of mercury, extinguished with oleate of mercury and mixed with prepared suet and benzoinated lard. (See p. 346.) It is used rather extensively in skin troubles, and also for destroying body vermin. For this latter purpose it is usually dispensed in diluted form.

UNGUENTUM HYDRARGYRI DILUTUM. Blue Ointment.

This is mercurial ointment U. S. P., so diluted with petrolatum as to yield a product containing 33 per cent. mercury.

The blending suggests the following problem:

How much mercurial ointment " $\frac{1}{2}$ " can be made from $\frac{1}{2}$ lb. of mercurial ointment " $\frac{1}{2}$ "?

Answer.—This is solved by alligation (p. 59), viz.:

$\frac{1}{2}$ diluted to $\frac{1}{3}$.

Take $\frac{1}{3}$ lb. of " $\frac{1}{2}$ " and dilute with petrolatum to make $\frac{1}{2}$ lb. of " $\frac{1}{3}$."

Hence $\frac{1}{3}$ lb. of " $\frac{1}{2}$ " makes $\frac{1}{2}$ lb. of " $\frac{1}{3}$."

Hence $\frac{1}{2}$ lb. of " $\frac{1}{2}$ " will make $\frac{\frac{1}{2}}{\frac{1}{3}} \times \frac{1}{2} = \frac{1}{4} \div \frac{1}{3} = \frac{1}{4} \times \frac{3}{1} = \frac{3}{4}$.

Hence $\frac{1}{2}$ lb. of " $\frac{1}{2}$ " will make $\frac{3}{4}$ lb. of " $\frac{1}{3}$."

If preferred, the problem may be worked as percentages, viz.:

50 per cent. diluted to 33 $\frac{1}{3}$ per cent.

33 $\frac{1}{3}$ lb. of 50 per cent. will make 50 lb. of 33 $\frac{1}{3}$ per cent.

Hence 0.5 lb. of 50 per cent. will make $\frac{0.5}{33\frac{1}{3}} \times 50$ lb. of 33 $\frac{1}{3}$ per cent.

Or $\frac{25}{33\frac{1}{3}} = \frac{75}{100} = \frac{3}{4}$ lb.

In the pharmacopœia of 1890 the 50 per cent. ointment was the only one official, and its official synonym was blue ointment. Most pharmacists dispensed over the counter in answer to calls for "blue ointment" the 33 per cent. article, and under the pure food laws of the several States such substitution of a weaker product for the official was a misdemeanor.

As the 33 per cent. article serves the domestic purposes as well as does the 50 per cent. ointment, the revision committee introduced the diluted ointment into the new pharmacopœia, giving it the English official name of blue ointment.

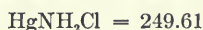
EMPLASTRUM HYDRARGYRI. Mercurial Plaster.

This preparation, containing 30 per cent. of mercury extinguished with oleate of mercury, and then diluted with lead plaster. (See p. 356.)

Emplastrum Ammoniaci cum Hydrargyri (U. S. P. 1890).—Plaster of ammoniac and mercury, which was formerly official, contains 18 per cent. of mercury, and is made by extinguishing the metal with oleate of mercury, and combining same with an acetic acid extract of ammoniac and lead plaster.

SALTS OF MERCURY

It will be recalled, in considering iron salts, the various ferrous compounds were first discussed, and these followed by the ferric compounds. In the case of the mercury compounds, however, we can obtain a better view of the subject by comparing the two classes of salts, the "ous" and the "ic" together; hence the two chlorides will be studied together, then the two iodides, and so on.

HYDRARGYRUM AMMONIATUM. Ammoniated Mercury.

It should contain not less than 78 per cent. nor more than 80 per cent. of metallic mercury.

Recipe.—Corrosive Mercuric Chloride, in powder, *one hundred grammes*. 100 Gm.
Ammonia Water,
Distilled Water, each, *a sufficient quantity*.

Dissolve the Corrosive Mercuric Chloride in *two thousand cubic centimeters* of warm Distilled Water, filter the solution, and allow it to cool. Pour the filtered liquid gradually, and with constant stirring, into *one hundred and fifty cubic centimeters* of Ammonia Water, taking care that the latter shall remain in slight excess. Collect the precipitate on a filter, and, when the liquid has drained from it as much as possible, wash it with a mixture of *four hundred cubic centimeters* of Distilled Water and *twenty cubic centimeters* of Ammonia Water. Finally, dry the precipitate between sheets of bibulous paper, in a dark place, at a temperature not exceeding 30° C. (86° F.), and keep it in well-stoppered bottles, protected from light.

White, pulverulent pieces, or a white, amorphous powder, without odor, and having an earthy, afterward styptic and metallic taste; permanent in the air.

Insoluble in water or in alcohol. By prolonged washing with water it is gradually decomposed, assuming a yellow color, and becoming converted into a basic salt. Readily soluble in warm hydrochloric, nitric, or acetic acid, and in a cold solution of ammonium carbonate. Also completely soluble in a cold solution of sodium thiosulphate, with the evolution of ammonia; when this solution is heated for a short time, red mercuric sulphide is separated, which, on protracted boiling, turns black.

At a temperature below a red heat Ammoniated Mercury is decomposed without fusion, and at a red heat it is wholly volatilized.

The salt should be soluble in hydrochloric acid without effervescence (absence of carbonate) and without leaving a residue (absence of mercurous salt).

Tests for Identity.—See Mercury, Ammonium, and Chlorides in Part V.

Impurities.—Carbonate, mercurous salts. See above. Foreign salts. See Mild Mercurous Chloride. Metals and arsenic. See Corrosive Mercuric Chloride.

Remarks.—This compound, commonly called *white precipitate*, is

made by the precipitation of ^athe solution of mercuric chloride with ammonia water, the equation being as follows:



Ammoniated mercury is an interesting illustration of how a complex formula can be altered. If we add hydrochloric acid (HCl) to ammonia water (NH_4OH), we get ammonium chloride (Formula A), as already explained on p. 483.

If, however, it is mercuric chloride (HgCl_2) that is added to ammonia water instead of hydrochloric acid, we get ammoniated mercury, NH_2HgCl , in which two of the hydrogen atoms of the ammonium chloride are replaced by one atom of mercury, with the valence II, as shown in Formula B.

FORMULA A.



FORMULA B.



Great care must be taken in the manufacture strictly to follow the pharmacopœial recipe. Several combinations of mercuric chloride with ammonia are known, and if the proportions of bichloride and ammonia water vary, some of these other than HgNH_2Cl may be formed. Again, if the diluted ammonia water is poured into the bichloride solution instead of the pharmacopœial direction of the reverse procedure, we obtain a double salt, $\text{NH}_2\text{HgClHgCl}_2$. Even use of exactly the pharmacopœial amount of wash liquid is imperative, as a large excess of water will convert the precipitate to the yellow $\text{NH}_2(\text{HgOHg})\text{Cl}$.

White precipitate, when treated with alcoholic solution of iodine, produces an explosion due to liberation of ammonia and nitrogen:



Ammoniated mercury is never used internally, being always applied in the form of an ointment. Such an ointment, containing 10 per cent. of ammoniated mercury, rubbed up with 50 Gm. melted white petrolatum and 40 Gm. hydrous wool-fat, is official under the name of *unguentum hydrargyri ammoniati*. (See p. 346.)

Sal Alembroth, $\text{Hg}(\text{NH}_4)_2\text{Cl}_2 \cdot 2\text{H}_2\text{O}$, a crystalline, water-soluble salt, made by dissolving mercuric chloride and ammonium chloride in boiling water, was formerly used in medicine.

Two chlorides of mercury are recognized by the pharmacopœia—the *mercuric chloride*, HgCl_2 , and the *mercurous chloride*, HgCl .

HYDRARGYRI CHLORIDUM CORROSIVUM. Corrosive Mercuric Chloride.



It should contain not less than 99.5 per cent. of pure Mercuric Chloride and be kept in well-stoppered bottles.

Heavy, colorless, rhombic crystals, or crystalline masses, odorless, and having an acid and persistent metallic taste; permanent in the air.

When in fine powder, it is soluble in 13 parts of water, 3 parts of alcohol, and in about 14 parts of glycerin at 25° C. (77° F.); soluble in 2 parts of boiling water and in 1.2 parts of boiling alcohol.

If 1 Gm. of finely powdered Mercuric Chloride be dissolved in 10 Cc. of alcohol or 20 Cc. of water, it should leave not more than 0.005 Gm. of residue.

It fuses at 265° C. (509° F.) to a colorless liquid, and at about 300° C. (572° F.) it volatilizes in dense, white vapors, leaving no appreciable residue.

The aqueous solution reddens blue litmus-paper, but becomes neutral to litmus-paper upon the addition of sodium chloride.

If to 0.5 Gm. of Mercuric Chloride, dissolved in 20 Cc. of water, 5 Cc. of hydrochloric acid be added, and the solution be completely saturated with hydrogen sulphide, allowed to stand for several hours in a well-corked flask until the precipitate has subsided, and then filtered, the filtrate should be colorless and leave no weighable residue upon evaporation (absence of many *foreign salts*).

If the precipitate obtained in the preceding test, after washing with about 100 Cc. of water and draining, be rinsed into a beaker with about 20 Cc. of water, and then 5 Cc. of stronger ammonia water added, and if after covering and digesting the mixture for about 15 minutes on a bath of boiling water, it be rinsed upon a filter and washed with a little water, the filtrate and washings after evaporating to dryness, moistening with 6 drops of nitric acid, and again drying, should not respond to the Modified Gutzeit's Test for *arsenic* (see p. 973). If the precipitated sulphide remaining upon the filter be treated with diluted nitric acid (1 in 4), warmed, and then filtered, the filtrate should leave a weighable residue upon evaporation and gentle ignition (limit of *foreign metals*).

Tests for Identity.—See Mercury Salts and Chlorides in Part V.

Impurities.—Foreign salts, arsenic, foreign metals, see above.

This is the violent poison called *corrosive sublimate*, and is made by heating a mixture of mercuric sulphate and sodium chloride in a retort. As a result, the mercuric chloride forms and sublimes, condensing in masses, while the sodium sulphate remains in the retort, as is shown in the following equation:



Mercuric chloride, or *bichloride of mercury*, as it is frequently called, occurs in the form of colorless, rhombic crystals or in crystalline masses. It is used internally as an alterative, and is frequently administered in combination with potassium iodide, when mercuric iodide results.

Mercuric chloride is one of the most valuable antiseptics. If it is applied to the skin in concentrated form, it is escharotic and caustic, and for this reason great care should be taken in the use of the alcoholic solution for local applications.

Bichloride of mercury is a virulent corrosive poison, and its antidote is albuminous matter, preferably the white of eggs, with which it forms a comparatively inert, insoluble albuminate of mercury. In lieu of white of eggs, milk can be used as an antidote. All these substances should, however, be followed by an emetic.

Dose.—3 milligrammes ($\frac{3}{10}$ grain).

HYDRARGYRI CHLORIDUM MITE. Mild Mercurous Chloride.



It should contain not less than 99.5 per cent. of pure Mercurous Chloride and be kept in dark amber-colored bottles.

A white, impalpable powder, becoming yellowish-white on being triturated with strong pressure, and showing only small, isolated crystals when viewed under a lens

having a magnifying power of one hundred diameters. It is odorless and tasteless and permanent in the air.

Insoluble in water, alcohol, or ether, and also in cold dilute acids.

When strongly heated, Mercurous Chloride is volatilized without fusion or the evolution of brown vapors, leaving no appreciable residue.

In contact with calcium hydroxide T.S., or with solutions of alkali hydroxides, or with ammonia water, the salt is blackened.

When heated with dried sodium carbonate in a dry glass tube, it yields a sublimate of metallic mercury.

If 1 Gm. of the salt be shaken with 10 Cc. of water or alcohol, and the mixture filtered, neither of the filtrates should respond to the Time-Limit Test for *heavy metals* (see p. 979), nor should any appreciable residue be left on evaporation (absence of *soluble impurities*).

If 2 Gm. of the salt be shaken with 20 Cc. of ether, filtered, the filtrate evaporated, and 10 Cc. of distilled water added, not more than a slight opalescence should result upon the addition of silver nitrate T.S. to 5 Cc. of the filtrate, and no change in color should be produced upon adding a few drops of ammonium sulphide T.S. to the remainder (absence of *mercuric chloride*).

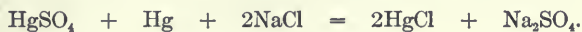
On heating a portion of the salt in a test-tube with potassium hydroxide T.S., it should not evolve ammonia; and if another portion be shaken with acetic acid and filtered, the filtrate should not be affected by hydrogen sulphide T.S. nor by silver nitrate T.S. (distinction from, and absence of, *ammoniated mercury*).

Tests for Identity.—Given above.

Impurities.—Heavy metals, soluble impurities, mercuric chloride, ammoniated mercury. Details above. Foreign salts, like corrosive sublimate test, except the substance, is dissolved in nitric acid. Arsenic and foreign metals; see Corrosive Sublimate.

Remarks.—Two origins of the synonym *calomel* have been suggested. Both agree that the word is derived from the Greek word *kalos*, meaning good, and *melas*, meaning black. Some explain these words (black, good) by saying that it means that calomel was good for black bile, while others claim that it is because of the fact that calomel blackens when treated with ammonia.

Calomel is made by a process similar to the manufacture of corrosive sublimate—*i. e.*, the mercuric sulphate is prepared by treatment of mercury with sulphuric acid, and to the mass sodium chloride is added, but before subliming there is also added an amount of mercury equal to the quantity originally used to make the mercuric sulphate. The mixture is then sublimed, as in the case of corrosive sublimate, for, be it remembered, calomel is sublimable, even as is the so-called "sublimate" (HgCl_2). The equation of manufacture is as follows:



The two chlorides, calomel and corrosive sublimate, afford a vivid picture of the possible immense alteration in therapeutic or physical condition that a slight chemical difference may produce.

Between the insoluble, comparatively harmless calomel, and the soluble and virulent poison, corrosive sublimate, the only chemical difference is that the former contains more mercury than does the latter. The quantity of mercury in each is best shown by the following estimation of percentage composition of the two salts:

CORROSIVE SUBLIMATE.

$$\text{Hg} = 1 \times 200 = 200$$

$$\text{Cl}_2 = 2 \times 35.5 = 71$$

$$\text{Molecular weight, HgCl}_2, 271$$

CALOMEL.

$$\text{Hg}_2 = 2 \times 200 = 400$$

$$\text{Cl}_2 = 2 \times 35.5 = 71$$

$$\text{Molecular weight, Hg}_2\text{Cl}_2, 471$$

From this it will be seen that while corrosive sublimate is $\frac{200}{771}$, or 73.4 per cent., mercury, calomel contains $\frac{400}{771}$, or 84.9 per cent., mercury.

Since calomel and corrosive sublimate are both sublimable and are prepared by similar processes, there is always a possibility of the calomel containing traces of corrosive sublimate, and for this reason the pharmacopœial test should be applied to all samples of calomel. This test consists in shaking the sample of calomel with ether (in which it is insoluble, but which will dissolve the bichloride), evaporation of the ethereal solution, taking up residue with water, and testing water solution for bichloride either with silver nitrate or ammonium sulphide.

A simpler, though not exact, test was provided in U. S. P. 1890, namely, that calomel is totally insoluble in water, whereas, if there is any bichloride present, it will readily dissolve in water. Therefore on mixing calomel with water, the water, on being filtered, will contain bichloride, if any be present in the calomel, and can be detected by treating with ammonia and hydrogen sulphide.

Mild mercurous chloride is a valuable hepatic stimulant and alterative. In former days very large doses of calomel were given, ten grains being considered an average dose. It has been discovered, however, that small doses frequently repeated act far better than single large doses. For this reason at present this chemical is usually administered in $\frac{1}{8}$ - to $\frac{1}{2}$ -grain doses every two hours. It must be remembered that if a patient who has taken calomel is permitted to consume food containing free acids, such as lemonade, etc., that form of mercurial poisoning called salivation ensues. Hence when calomel is administered, the patient should always be warned to avoid the use of such acids until the chemical has completely acted.

A favorite method of administration of calomel is in combination with an alkali, preferably sodium bicarbonate. Large quantities of this combination are consumed in the southern States, and it might be stated that pills containing $2\frac{1}{2}$ grains each of calomel and sodium bicarbonate have enjoyed considerable vogue under the name of *Dr. Stone's Little Giant Pills*. Two of these were considered a dose. It has been claimed that an alkali acting on the calomel has the effect of converting some of it into the bichloride of mercury, and to discover the truth regarding this matter experiments were made by combining calomel with sugar, with milk-sugar, with magnesium carbonate and sodium bicarbonate, and testing the mixture, after an appropriate period, for bichloride of mercury. It was found that in none of these mixtures was bichloride found after standing twenty-four hours. No bichloride was found in the mixtures of calomel with magnesia, magnesium carbonate, or sugar after standing three months. Traces of bichloride were found in the mixtures of calomel with milk-sugar or sodium bicarbonate after three months. While a large amount was found after three months in the mixture of calomel, sodium bicarbonate, and sugar. It was also found that combining calomel with salt tended to the formation of bichloride of mercury.

This experiment shows the advisability of using freshly prepared doses of calomel and soda, and warns us against dispensing tablets of calomel and soda bought in the open market. On the other hand, it might be added that some physicians desire and expect the formation

of minute traces of mercuric chloride or oxide when calomel and soda are prescribed, and that there is an idea that if the two ingredients be thoroughly triturated, such a change will take place. For this reason some physicians specify in their prescriptions thorough trituration, so that the expected bichloride may be formed.

Dose.—Laxative, 125 milligrammes (2 grains). Alterative, 65 milligrammes (1 grain).

Hydrargyri Cyanidum (U. S. P. 1890).—Mercuric cyanide, HgCN . This chemical is made by passing hydrocyanic acid gas into a liquid in which finely divided mercuric oxide is suspended. The hydrocyanic acid used is generally produced by treatment of potassium ferrocyanide with sulphuric acid.

Mercuric cyanide occurs in colorless prismatic crystals, turning dark on exposure to air, and is used as an alterative in syphilis, administered in doses of from 4 to 8 milligrammes.

Two iodides of mercury are recognized by the pharmacopœia—*mercuric iodide*, HgI_2 , and *mercurous iodide*, HgI .

HYDRARGYRI IODIDUM RUBRUM. Red Mercuric Iodide.



It should contain not less than 99.5 per cent. of pure Mercuric Iodide.

Recipe.—Corrosive Mercuric Chloride, *forty grammes*..... 40 Gm.
Potassium Iodide, *fifty grammes*..... 50 Gm.
Distilled Water, *a sufficient quantity*.

Dissolve the Corrosive Mercuric Chloride and the Potassium Iodide, each, in *eight hundred cubic centimeters* of Distilled Water, and filter the solutions separately. Pour both solutions, simultaneously and in a thin stream, with constant and very active stirring, into *two thousand cubic centimeters* of Distilled Water. When the precipitate has subsided, decant the supernatant liquid, collect the precipitate on a filter, and wash it with cold Distilled Water, until the washings give not more than a slight opalescence with silver nitrate test solution. Finally, dry it in a dark place, between sheets of bibulous paper, at a temperature not exceeding 40°C . (104°F .), and keep it in well-stoppered bottles, protected from light.

A scarlet-red, amorphous powder, odorless and tasteless; permanent in the air.

Almost insoluble in water; soluble in 116 parts of alcohol, 85 parts of ether, and 1340 parts of chloroform at 25°C . (77°F .); soluble in 15 parts of boiling alcohol; also soluble in solutions of the soluble iodides, mercuric chloride, sodium thiosulphate, and hot solutions of the alkali chlorides.

When heated to about 150°C . (302°F .) the salt becomes yellow, but again assumes a red color on cooling; at 253°C . (487.4°F .) it fuses to a dark yellow liquid, which, on cooling, forms a yellow, crystalline mass, and at higher temperatures is finally volatilized, leaving no appreciable residue.

On heating the salt with potassium hydroxide T. S., and adding a little sugar of milk, metallic mercury is precipitated.

A saturated solution of Mercuric Iodide in hot alcohol should, after cooling, be colorless; and when diluted with an equal volume of water the solution should not redden blue litmus-paper (absence of *mercuric chloride*).

Tests for Identity.—Given above.

Impurities.—Mercuric chloride. See above. Excess of soluble chlorides or iodides. See Part V.

Remarks.—This substance is made by the double decomposition of mercuric chloride and potassium iodide, avoiding excess of either reagent, as mercuric iodide is soluble in solutions of either chemical. This reaction takes place in many prescriptions, as the combination

is a favorite one in syphilis, and, as in all such prescriptions, the iodide is largely in excess, the precipitate formed invariably redissolves. A redissolved solution of the red iodide in potassium iodide is called "*Mayer's reagent*," the proportions being 13.55 Gm. mercuric chloride, 49.8 Gm. potassium iodide, and water to make 1000 Cc. As cited on p. 876, this is an alkaloidal precipitant, so look out for prescriptions calling for the corrosive sublimate, potassium iodide, and some tincture, or other preparation containing alkaloids, as the Mayer's reagent formed will surely precipitate the alkaloid.

The red mercuric iodide is used as an alterative in the case of syphilis.

As mentioned above, the physiologic effects of red mercuric iodide are obtained by prescribing a combination of potassium iodide with bichloride of mercury. Red mercuric iodide is a constituent of Donovan's solution. (See p. 184.)

Dose.—3 milligrammes ($\frac{1}{20}$ grain).

HYDRARGYRI IODIDUM FLAVUM. Yellow Mercurous Iodide.

HgI = 324.40

It should contain not less than 99.5 per cent. of pure Mercurous Iodide.

Recipe.—Mercury, fifty grammes. 50 Gm.
Nitric Acid,
Potassium Iodide,
Distilled Water, each, a sufficient quantity.

Mix twenty cubic centimeters, each, of Nitric Acid and Distilled Water, and, when the liquid is cold, pour it upon the Mercury contained in a glass beaker. Set the mixture aside in a dark place, and keep it at a temperature between 25° and 30° C. (77° and 86° F.), with occasional agitation, until the reaction ceases and a little mercury still remains undissolved. Separate the crystals of mercurous nitrate, which will have formed, from the mother-liquor, allow them to drain in a glass funnel, and dry them on bibulous paper, in a dark place. When the salt is dry, dissolve forty grammes of it in six hundred and fifty cubic centimeters of Distilled Water, to which six cubic centimeters of Nitric Acid have previously been added. Having prepared a solution of sixteen grammes of Potassium Iodide in thirty-two cubic centimeters of Distilled Water, slowly pour the solution of Potassium Iodide into the solution of Mercurous Nitrate, with constant stirring, which should be continued for fifteen minutes, allow the precipitate to subside, decant the supernatant liquid, and wash the precipitate by decantation with ten successive portions of five hundred cubic centimeters each of Distilled Water. Finally, transfer the precipitate to a filter, and dry it between sheets of bibulous paper, in a dark place, at a temperature not exceeding 40° C. (104° F.), and keep it in dark amber-colored vials, with the least possible exposure to light.

Instead of weighing off forty grammes of the Mercurous Nitrate as above directed, the whole of the crystallized salt may be taken and the amount of Potassium Iodide, etc., adjusted to the proportions given above.

A bright yellow, amorphous powder, odorless and tasteless. By exposure to light it becomes darker, in proportion as it undergoes decomposition into metallic mercury and mercuric iodide.

Almost insoluble in water and wholly insoluble in alcohol or ether.

When slowly and moderately heated, it assumes at first an orange and then a red color, becoming yellow again on cooling. When quickly and strongly heated, it is at first partially decomposed into mercury and mercuric iodide, and finally is volatilized, leaving not more than 0.05 per cent. of residue.

When Mercurous Iodide is heated with sulphuric acid and a little manganese dioxide, vapor of iodine is evolved.

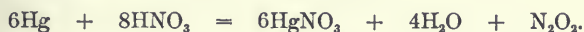
In contact with a solution of potassium iodide, the salt is decomposed into mercuric iodide, which dissolves, and metallic mercury, which remains undissolved.

If 0.5 Gm. of the salt be shaken with 10 Cc. of alcohol and the mixture allowed to stand and then filtered, a portion of the perfectly clear filtrate should be scarcely affected by hydrogen sulphide T.S., nor should it produce more than a very faint, transient opalescence when dropped into water; and if 5 Cc. of the filtrate be evaporated on a white porcelain surface, not more than a very faint red stain should remain (absence of more than traces of *mercuric iodide*).

Tests for Identity.—Given above.

Impurities.—Mercuric iodide. See above.

Remarks.—This chemical was called the *green iodide of mercury* in the pharmacopœia of 1880. Its manufacture, as directed by the pharmacopœia (eighth revision) is by dissolving mercury in a sufficient quantity of nitric acid to form a solution of mercurous nitrate, the equation being as follows:



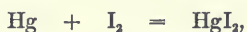
The resulting solution of mercurous nitrate is then treated with a molecular quantity of potassium iodide, when potassium nitrate and mercurous iodide result, the latter being precipitated in the form of yellow, crystalline powder, the equation being as follows:



This product is not the same as the green iodide of the pharmacopœia of 1880, which was made by the direct union of mercury with iodine, triturating the two in the presence of alcohol. The equation of this reaction was supposed to be as follows:



but, as a matter of fact, it partly runs as follows:



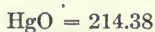
with the formation of mercuric iodide. This mercuric iodide was removed by the process of the pharmacopœia of 1880 by washing the mixture with alcohol.

Mercurous iodide, like mercuric iodide, is used as an alterative, but is a much milder preparation, as is shown by the doses.

Dose.—10 milligrammes ($\frac{1}{4}$ grain).

While the two iodides are mercurous and mercuric respectively, both *oxides of mercury* are “ic” salts, having the formula HgO , and giving an interesting example of physical difference of two bodies that are chemically identical. Their mutual identity is proved by the fact that red oxide, when finely triturated, turns yellow, while the yellow, on heating, is converted into the red.

HYDRARGYRI OXIDUM FLAVUM. Yellow Mercuric Oxide.



It should contain not less than 99.5 per cent. of pure Yellow Mercuric Oxide.

A light orange-yellow, amorphous, heavy, impalpable powder; odorless and having a somewhat metallic taste; permanent in the air, but turning darker on exposure to light.

Almost insoluble in water, insoluble in alcohol, but readily and completely soluble in diluted hydrochloric or nitric acid, forming colorless solutions.

When moderately heated, Yellow Mercuric Oxide assumes a red color. At a red heat it is completely decomposed into oxygen and metallic mercury, and is finally volatilized, leaving not more than 0.1 per cent. of residue.

When moistened with hot water, Yellow Mercuric Oxide should not turn red litmus-paper blue.

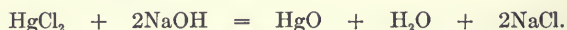
If 0.5 Gm. of Yellow Mercuric Oxide be digested on a water-bath, for fifteen minutes, with a solution of 1 Gm. of oxalic acid in 10 Cc. of water, it will be converted into white mercuric oxalate (distinction from *red mercuric oxide*).

Recipe and details of manufacture in Part VII.

Tests for Identity.—Given above.

Impurities.—Red mercuric oxide. See above. Excess of chlorides. See Part V. Foreign salts, metals, and arsenic, as in testing corrosive sublimate.

Remarks.—Yellow mercuric oxide is made by treating bichloride of mercury with the requisite amount of sodium hydrate, as shown in the following equation:



As a result, mercuric oxide is precipitated, leaving in solution sodium chloride, the last traces of which are removed from the mercuric oxide by washing with water.

Yellow mercuric oxide is used chiefly externally, and that in the form of a 10 per cent. ointment (see p. 347), or of a 25 per cent. solution in oleic acid. (See Oleate of Mercury on p. 211.)

HYDRARGYRI OXIDUM RUBRUM. Red Mercuric Oxide.

HgO = 214.38

It should contain not less than 99.5 per cent. of pure Red Mercuric Oxide and should be kept in well-stoppered bottles, protected from light.

Heavy, orange-red, crystalline scales, or a crystalline powder, becoming more yellow the finer it is divided; odorless and having a somewhat metallic taste; permanent in the air.

Almost insoluble in water, insoluble in alcohol, but readily and completely soluble in diluted hydrochloric or nitric acid, forming colorless solutions.

When heated to about 400° C. (752° F.), Red Mercuric Oxide becomes dark violet or almost black, but assumes its original color on cooling. At a red heat it is completely decomposed into oxygen and metallic mercury, and is finally volatilized, leaving no appreciable residue.

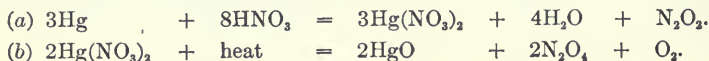
If 0.5 Gm. be digested on a water-bath with a solution of 1 Gm. of oxalic acid in 10 Cc. of water, it will not change color within two hours (distinction from *yellow mercuric oxide*).

Tests for Identity.—See above.

Impurities.—Yellow oxide. See above. Nitrates and excess of chlorides. See Part V. Foreign salts, metals, and arsenic, as in testing corrosive sublimate.

Remarks.—Red mercuric oxide is called "*red precipitate*," but is no longer prepared by precipitation, the name being a souvenir of alchemistic days, when it was made "upward precipitation," being formed in mercury kept near its boiling-point for weeks at a time.

It is now made by treating mercury with nitric acid (equation *a*) and heating the resulting mercuric nitrate to redness (equation *b*):



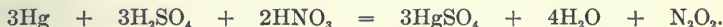
It will be seen from the above equations that the nitrate is dissociated into mercuric oxide, N_2O_4 , and oxygen, and experience has shown that the two gases are capable of converting into mercuric oxide an amount of mercury equal to the amount of the nitrate used originally. It will be noted that the pharmacopœia gives no directions for the manufacture of this compound by the pharmacist, although it does direct the preparation of the yellow mercuric oxide. The reason for the omission of the recipe for the red mercuric oxide is because its manufacture is scarcely practical on a small scale, it being found that at the heat necessary to convert the nitrate into the oxide a very large amount of mercury is lost by volatilization.

Red mercuric oxide is used almost entirely externally, and that chiefly in the form of an ointment for granulated eyelids.

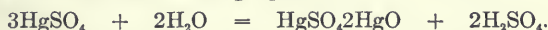
The pharmacopœia recognizes a 10 per cent. ointment in which the red oxide has been carefully levigated with water, and then rubbed with a hydrous wool-fat and petrolatum. (See p. 347.)

Hydrargyri Subsulphas Flavus (U. S. P. 1890).—The formerly official *yellow mercuric subsulphate* is a basic salt. As already explained on p. 561, "sub" salt is a combination of a metallic salt with its oxide, and in the case under consideration we find the relationship clearly shown in the symbolic formula, $HgSO_4(HgO)_2$. It is called *Turpeth mineral*, on account of its resemblance to the powdered turpeth root, the root of *Ipomœa turpethum*, which was formerly used in medicine.

Turpeth mineral is made by dissolving mercury in a mixture of sulphuric acid and nitric acids, whereby the soluble mercuric sulphate is formed, as shown in the following equation:



This solution of mercuric sulphate is poured into a large quantity of water, whereby the original white mercuric sulphate is converted into the yellow mercuric subsulphate, as shown in the following equation:



Turpeth mineral is an alternative, possessing, however, such irritating qualities as to render its general use unsafe. It is administered in doses of from 16 to 30 milligrammes.

Mercuric nitrate, $Hg(NO_3)_2$, is not recognized by the pharmacopœia as a chemical itself, but two preparations containing this body are given official recognition.

LIQUOR HYDRARGYRI NITRATIS. Solution of Mercuric Nitrate.

A liquid which should contain about 60 per cent. of Mercuric Nitrate [$Hg(NO_3)_2 = 321.64$] and about 11 per cent. of free nitric acid.

<i>Recipe</i> .—Red Mercuric Oxide, forty grammes.....	40 Gm.
Nitric Acid, forty-five grammes.....	45 Gm.
Distilled Water, fifteen grammes.....	15 Gm.
To make one hundred grammes..	100 Gm.

Mix the Nitric Acid with the Distilled Water and dissolve the Red Mercuric Oxide in the mixture. Keep the product in glass-stoppered bottles.

A clear, nearly colorless, heavy liquid, having a faint odor of nitric acid and a strongly acid reaction.

Specific gravity: about 2.086 at 25° C. (77° F.).

On evaporating a few drops of the Solution in a porcelain dish, a white residue is left, which, on being heated, becomes successively yellow, red, and brown, and is finally completely volatilized.

On a bright surface of copper the Solution deposits a coating of metallic mercury.

Tests for Identity.—See Mercury and Nitrates in Part V.

Impurities.—Mercurous salt (no precipitation nor cloudiness on adding water or diluted hydrochloric acid).

Remarks.—This official is made by dissolving 40 Gm. of mercuric oxide in a mixture of 45 Gm. of nitric acid and 15 Gm. of distilled water. It is one of the heaviest of the official solutions, being over twice as heavy as water (sp. gr., 2.086).

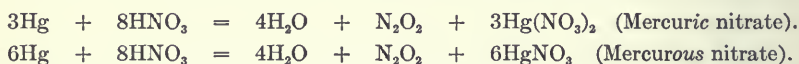
Solution of mercuric nitrate is used entirely locally as a corrosive in syphilitic sores.

UNGUENTUM HYDRARGYRI NITRATIS. Mercuric Nitrate Ointment.

Recipe and details of manufacture in Part VII.

The *ointment of mercuric nitrate* (called *citrine ointment*) is made by dissolving mercury in nitric acid, and, at the same time, heating lard to 105°C ., adding thereto a portion of the nitric acid. When the nitric acid is added to the heated lard, a rather violent chemical action takes place, with the conversion of the oleate of glyceryl, which is the chief constituent of the lard, into elaidin. (See p. 727.)

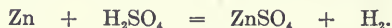
Mercury is dissolved in the rest of the nitric acid, forming mercuric nitrate, and it might be said in passing that when mercury is dissolved in nitric acid we get mercurous or mercuric nitrate, according to the relative proportions of mercury and nitric acid employed:



That the proportions of mercury and nitric acid used in the official recipe yield mercuric nitrate is shown by the molecular weight calculations relating to citrine ointment found in Practical Work (p. 1137).

It may be well to sum up here the effect of various acids on metals.

Hydrochloric and other haloid acids and diluted sulphuric acid give, with metals, the appropriate salt, *with the evolution of hydrogen*. Example:



Nitric acid, either concentrated or diluted, and *concentrated sulphuric acid*, give with metals the appropriate salt, with the *evolution of* N_2O_2 or N_2O , in the case of nitric acid, and of SO_2 in the case of sulphuric acid. Examples of nitric acid are given just above. Of concentrated sulphuric acid, see Copper Sulphate (p. 590).

Citrine ointment (from *citrus*, the lemon) has, as the name suggests, a bright yellow color when freshly prepared; on standing, however, it assumes a brown tint. The manufacture of this compound is one calling for considerable skill in order to obtain a product of exactly the right color and consistence and of the same quality. The quality of lard oil found in the market was so variable that, in revising the pharmacopœia, the lard oil of U. S. P. 1890 was replaced by the more reliable lard.

Citrine ointment is a valuable remedy for certain forms of skin trouble, such as tetter and ring-worm.

An unofficial salt of mercury worthy of consideration is *mercuric sulphide*, HgS , which was recognized by the pharmacopœia of 1880.

This substance is the artificial *cinnabar*, and is made by the sublimation of the black sulphide. The latter, formerly called *Æthiops mineral*, has been mentioned as the precipitate formed by passing hydrogen sulphide through solutions of mercuric salts, but is made commercially by fusing sulphur with mercury. Both sulphides have the formula HgS , the only difference being that the red is crystalline, while the black is amorphous. Cinnabar is practically never used internally.

SILVER

Symbol, Ag. Atomic weight, approximately 108

Silver occurs occasionally in the form of the pure metal, but its most abundant ores are silver sulphide, which is called *silver glance*, and the chloride, or *horn silver*. A very large quantity of silver is obtained from the silver-bearing galena. (See p. 597.)

Silver is one of the ancient metals, reference to it being found in the earliest writings. The Latin word *argentum* is derived from the Greek for silver, *arguros*, which is in turn derived from the Greek word *argos*, meaning white. It is worth the matter of passing interest that in several languages the word silver is similar to the word white, and we know that, in this country, we sometimes speak of silver as the white metal, in contradistinction to gold, "the yellow metal."

The *metallurgy* of silver is interesting and important, and can be roughly divided into four groups. The first include the wet extraction processes. In one of these the sulphides (galena or silver glance) is roasted with sodium chloride, thus changing the silver to silver chloride. The roasted mass is then ground, placed in appropriate hoppers, percolated with an aqueous solution of sodium thiosulphate, in which the chloride is soluble; sodium sulphide is added to the percolate, causing the precipitation of silver sulphide.

The pure silver is obtained by roasting the sulphide, while the thiosulphate is recovered from the filtrate and used again.

Another method is by carefully roasting the ores, whereby the silver sulphide contained therein is converted into sulphate, and this is extracted by percolation of the roasted mass with water. Metallic silver is separated from the percolate by use of metallic copper.

The second group of processes are those where *amalgamation* is employed. In these the ore is roasted with salt, changing the silver sulphide found therein to silver chloride. This is reduced with iron or with copper sulphate to metallic silver, which forms an amalgam with mercury. This amalgam is separated from the solid matter and is heated in a retort, when the mercury distils, leaving a residue of metallic silver.

The third group of processes are those where *cupellation* is employed, and is applied to ores containing lead and silver. Such ores are roasted in furnaces with free exposure to air, the result being the liberation of metallic silver and the conversion of lead into litharge, which is skimmed off. Lastly, *electrolytic separation* of silver from copper-bearing ores is now being employed.

Silver is a brilliant white metal, malleable, ductile, soluble in nitric acid, and but sparingly soluble in sulphuric acid. While in the molten condition it has a great affinity for oxygen, absorbing no

less than twenty-two times its volume of that gas. On cooling, it parts with all its oxygen, and frequently with explosive violence. This property makes the melting of silver a process requiring considerable care. The silver used in coins consists of 90 per cent. of silver and 10 per cent. of copper, it being so admixed because pure silver is too soft to be used alone.

Silver is called a "noble metal," partly because it costs more than the common metals already considered, and chiefly because it withstands the oxidizing action of the atmosphere. This property it shares with gold and platinum and is noted in a lesser degree in aluminum and nickel.

The tests for silver are as follows:

Hydrochloric acid produces a precipitate of white silver chloride, insoluble in nitric acid, but soluble in ammonia water. Hydrogen sulphide or ammonium sulphide produces a black precipitate of silver sulphide, while potassium hydroxide precipitates silver from its soluble salts in the form of a brown silver oxide.

The oxides of silver which are known are Ag_2O , Ag_2O_2 , and Ag_4O . Of these, the first is the only one of sufficient importance to merit more than passing notice. It is recognized by the pharmacopœia, and in constitution is analogous to sodium oxide, Na_2O , exhibiting silver with valence 1.

The official salts of silver are:

Silver cyanide. At least 99.9 per cent. absolute AgCN .

Silver nitrate. At least 99.9 per cent. absolute AgNO_3 .

Fused silver nitrate. At least 94.8 per cent. absolute AgNO_3 .

Mitigated silver nitrate. At least 33.3 per cent. absolute AgNO_3 .

Silver oxide. At least 99.8 per cent. absolute Ag_2O .

In considering these, the nitrate will be the first taken up, since from it all the other official silver compounds are made.

ARGENTI NITRAS. Silver Nitrate.

$\text{AgNO}_3 = 168.69$

It should contain not less than 99.9 per cent. of pure Silver Nitrate and should be kept in dark amber-colored vials, protected from light.

Colorless, transparent, tabular, rhombic crystals, becoming gray or grayish-black on exposure to light in the presence of organic matter; without odor, but having a bitter, caustic, and strongly metallic taste.

Soluble in 0.54 part of water, and in 24 parts of alcohol at 25°C . (77°F .); in 0.1 part of boiling water and in 5 parts of boiling alcohol.

When heated in a porcelain crucible to about 200°C . (392°F .) the salt melts, forming a faintly yellow liquid, which, on cooling, congeals to a pure white, crystalline mass. At a higher temperature it is gradually decomposed with evolution of nitrous vapors.

An aqueous solution of the salt is neutral to litmus-paper, and yields, with hydrochloric acid, a white precipitate, which is readily dissolved by ammonia water, the liquid not acquiring a blue color (absence of *copper*).

If a portion of an aqueous solution (1 in 10) be completely precipitated by hydrochloric acid, filtered, and the filtrate evaporated to dryness, no residue should be left (absence of *foreign salts*).

Tests for Identity.—Given above.

Impurities.—Copper, foreign salts. See above. Lead. See Part V.

Assay.—Details in Part V.

Remarks.—This salt is made by dissolving silver coin in nitric acid, evaporating the solution to dryness, fusing, adding water to the fused mass, and filtering the silver nitrate solution from the copper oxide residue.

Silver coin is far more accessible than pure silver, hence is employed in making the nitrate. But in this process not only does the silver unite with the acid to form the nitrate, but, likewise, the copper forms copper nitrate, and, of course, a silver nitrate containing copper nitrate would not answer. The copper is removed in a very ingenious way. Silver nitrate melts unchanged at 200°C. , and at that temperature copper nitrate is decomposed into the gaseous oxides of nitrogen and into copper oxide, which remains behind, an insoluble mass. The fused mass is treated with water, in which the silver nitrate dissolves, leaving copper oxide behind.

It will be noted, from the pharmacopœial description given above, the solution of silver nitrate turns black on exposure to light. This is due to the separation of metallic silver, under the reducing action of sunlight. All silver salts are very sensitive to the action of light; most of all, silver bromide, and the blackening of silver bromide by the influence of light constitutes the foundation of the art of photography.

Silver nitrate is a very valuable remedy, used both externally and internally. It is used internally in cases of gastritis and diarrhea in small doses, while large quantities act as a caustic and escharotic. If given in an overdose, the antidote is sodium chloride, which produces a mild and comparatively inert silver chloride. Externally, it is of considerable value as a caustic, but the application should be made with great care. For external use two other forms of silver nitrate are provided by the pharmacopœia, the fused and the diluted.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

ARGENTI NITRAS FUSUS. Molded Silver Nitrate.

It should contain not less than 94.8 per cent. of pure Silver Nitrate.

Recipe.—Silver Nitrate, one hundred grammes..... 100 Gm.
Hydrochloric Acid, four grammes..... 4 Gm.

To the Silver Nitrate, contained in a porcelain dish, add the Hydrochloric Acid, and melt the mixture at as low a temperature as possible. Stir well, and pour the melted mass into suitable molds. It should be kept in dark amber-colored vials, protected from light.

A white, hard solid, generally in the form of pencils or cones of a fibrous fracture, becoming gray or grayish-black on exposure to light in the presence of organic matter; odorless and having a bitter, caustic, and strongly metallic taste.

Soluble, with the exception of about 5 per cent. of silver chloride, in 0.54 part of water and in 24 parts of alcohol at 25°C. (77°F.); in 0.1 part of boiling water and 5 parts of boiling alcohol. The portion left undissolved by water should be completely soluble in ammonia water.

A clear, aqueous solution of Molded Silver Nitrate, decanted from the insoluble portion, should be neutral to litmus-paper, and should respond to the tests of identity and purity stated under *Argenti Nitrās*.

Tests for Identity.—See above.

Impurities.—As in silver nitrate.

Assay.—Details in Part V.

Remarks.—Fused silver nitrate is made by melting silver nitrate in a porcelain capsule, adding thereto 4 per cent. of hydrochloric acid, and pouring the melted mixture into suitable molds. The hydrochloric acid used in this case is for the purpose of rendering the silver nitrate tougher and less brittle by the formation of a small quantity of silver chloride.

The melted silver nitrate is called *lunar caustic* (from *luna*, Latin for moon), and the application in this case is an interesting relic of alchemistic nomenclature. The alchemists, the forerunners of the modern chemists, assigned to the commoner metals the names of the heavenly bodies and of the gods of Roman mythology. Thus, gold was the sun; silver, the moon (*luna*); iron was Mars; copper was Venus; lead was Saturn; while quicksilver was, as it is now, mercury. In old names that are still used,—like “*boule de Mars*” (iron and potassium tartrate), “*acetum Saturni*” (solution of lead subacetate), and “*lunar caustic*,”—we find these terms still existing.

Molded silver nitrate is also called *lapis infernalis* (*stone of Hell*).

ARGENTI NITRAS MITIGATUS. Mitigated Silver Nitrate.

[ARGENTI NITRAS DILUTUS, PHARM. 1890]

It should contain not less than 33.3 per cent. of pure Silver Nitrate.

Recipe.—Silver Nitrate, thirty grammes. 30 Gm.
Potassium Nitrate, sixty grammes. 60 Gm.

Melt the salts together in a porcelain crucible, at as low a temperature possible, as stirring the melted mass well until it flows smoothly. Then pour it into suitable molds. It should be kept in dark amber-colored vials, protected from light.

A white, hard solid, generally in the form of pencils or cones of a finely granular fracture, becoming gray or grayish-black on exposure to light in the presence of organic matter; odorless and having a caustic, metallic taste.

Its aqueous solution is neutral to litmus-paper.

Each of its constituents retains the solubility in water and in alcohol stated respectively under *Argenti Nitrates* and *Potassii Nitrates*.

Tests for Identity.—See Silver, Potassium, and Nitrates in Part V.

Impurities.—Copper, lead, and bismuth (no blue color nor turbidity on adding excess of ammonia to aqueous solution).

Assay.—Details in Part V.

Remarks.—This chemical is made by melting together 30 parts of silver nitrate and 60 parts of potassium nitrate. As silver nitrate is a very powerful caustic, it is sometimes found wise to apply it in this diluted form.

ARGENTI CYANIDUM. Silver Cyanide.

$\text{AgCN} = 132.96$

It should contain not less than 99.9 per cent. of pure Silver Cyanide, corresponding to 80.48 per cent. of metallic silver. It should be kept in dark amber-colored vials, protected from light.

A white powder, without odor or taste, permanent in dry air, but gradually turning brown on exposure to light.

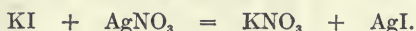
Insoluble in water, alcohol, or cold nitric acid, but soluble in boiling nitric acid with evolution of hydrocyanic acid; also soluble in ammonia water, sodium thio-sulphate T.S., and potassium cyanide T.S.

When heated in a porcelain crucible the salt fuses, gives off cyanogen gas, and, on ignition, leaves a residue of metallic silver amounting to 80.48 per cent. of its original weight.

Remarks.—This chemical is made by passing hydrocyanic gas through a solution of silver nitrate. It can also be made by treating potassium cyanide with silver nitrate.

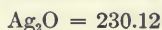
It is official only because it affords a simple method of extemporaneous preparation of diluted hydrocyanic acid. This acid is quite unstable, and should be fresh when dispensed. This rule is easily followed if the acid is made from silver cyanide and hydrochloric acid, the hydrocyanic acid that is formed remaining in solution, while the silver chloride separates out and can be filtered off.

Argenti Iodidum (U. S. P. 1890).—*Silver iodide.* This salt is made by combining a solution of potassium iodide and silver nitrate and collecting the precipitated silver iodide, as shown in the following equation:



This is a heavy yellow powder, and is used as an alterative in syphilis, in doses of 0.03 to 0.12 Gm. ($\frac{1}{2}$ to 4 grains).

ARGENTI OXIDUM. Silver Oxide.



It should contain 99.8 per cent. of pure Silver Oxide, corresponding to not less than 92.9 per cent. of pure metallic silver, and should be kept in dark amber-colored vials. Silver Oxide should not be triturated with readily oxidizable or combustible substances, and should not be brought in contact with ammonia.

A heavy, dark brownish-black powder, liable to reduction by exposure to light, odorless, and having a metallic taste.

Very slightly soluble in water, to which it imparts an alkaline reaction; insoluble in alcohol; it is readily and completely soluble in nitric acid without effervescence (absence of *carbonate* or *chloride*).

When heated in a porcelain crucible to about 250° to 300° C. (482° to 572° F.), it is rapidly decomposed, with the evolution of oxygen, leaving a residue of metallic silver.

The solution of the Oxide in nitric acid should be colorless, and should respond to the reactions and tests stated under *Argenti Nitras*.

If 0.5 Gm. of the Oxide be ignited in a porcelain crucible, it should yield not less than 0.464 Gm. of pure metallic silver.

Tests for Identity. Given above.

Impurities.—Carbonate, chloride, see above. Copper, lead, foreign salts. See Silver Nitrate.

Assay.—See above.

Remarks.—Silver oxide is prepared by precipitating silver nitrate solution with potassium hydroxide solution, as shown in the following equation:



Silver oxide must be carefully handled, owing to its energetic oxidizing action, hence the pharmacopœial warning given above.

The only way safely to make pills of silver oxide is by using petroleum mass or kaolin and vaselin as excipient. (See p. 315.) Also beware of bringing it in contact with ammonia, for the two will form the dangerous explosive, fulminating silver.

Silver oxide is one of the most valuable compounds of silver for internal use, it being far less caustic than any of the other silver compounds.

Dose.—65 milligrammes (1 grain).

PART IV

ORGANIC CHEMISTRY

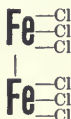
CHAPTER XXXV

INTRODUCTION

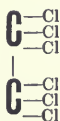
ORGANIC chemistry is the study of the carbon compounds, the adjective "organic" being a survival of the earlier days of chemical thought, when it was supposed that all substances peculiar to animal and vegetable economy—such bodies as those about to be discussed—were produced by the action of a mysterious influence called the force of life—"vis vitæ." Although this vis vitæ theory was exploded in 1828 by Woehler's synthesis of urea (p. 749), the name "organic chemistry" is still applied to the carbon compounds.

Essential to an organic compound are the carbon atoms it contains, and it is interesting to note that in all the numberless derivatives of carbon (some 112,000 now known to the practice) these carbon atoms always show the valence IV, just as do the carbon atoms in the carbonates discussed in previous chapters.

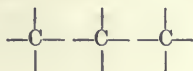
The reason why carbon forms so many compounds is because it possesses, to a far greater extent than any other element, the property of using some of its bonds for linking itself to adjoining carbon atoms. That one atom of an element is capable of attaching itself to another atom of the same element has been already shown, for example, in the formula Fe_2Cl_6 (p. 548), the graphic formula showing that two atoms of iron are directly attached to each other. Likewise in Hg_2Cl_2 the two mercury atoms are attached to each other by their own bonds. Returning to the question of ferric chloride, it will be remembered that its formula was explained by the statement that we had iron with the valence IV, and with a bond of each iron atom used for attaching itself to the other, as shown in graphic formula:



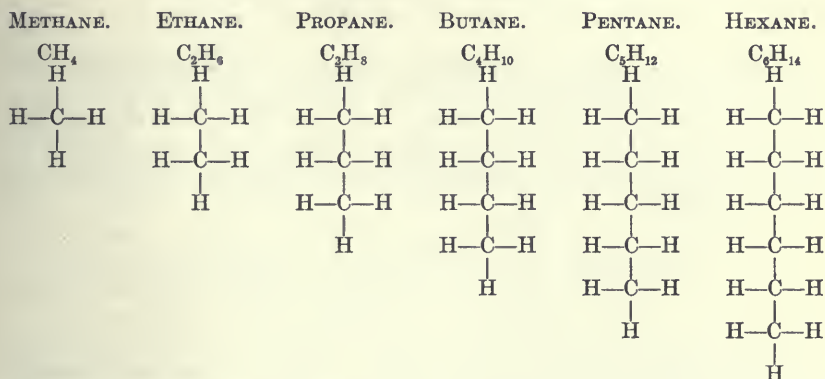
Exactly analogous to ferric chloride is the formula of ethane, that carbon compound containing two atoms, and, as a matter of fact, there is known a body C_2Cl_6 , exactly analogous to Fe_2Cl_6 :



These two carbon atoms, however, can link on a third carbon atom, making a chain of three carbon atoms, which can be graphically represented as:



And in this way there can be devised chains containing four, five, or six carbon atoms, making the following series:



This series can be continued almost indefinitely, similar chains containing 60 carbon atoms being known.

These compounds are composed only of carbon and hydrogen, and are called *hydrocarbons*. They all belong to a similar set or series, and the members of such a series are called *homologues*. In this series of homologues the carbon atoms are linked into a long string or chain, and the series is called the *chain series* of hydrocarbons. Because these hydrocarbons and their derivatives are found in fats, the series is sometimes called the *fatty series*.

Among the members of the series there exists a mathematic relationship between the relative number of carbon and hydrogen atoms.

This relationship can be expressed in the mathematic formula $\text{C}_n\text{H}_{2n+2}$, which means twice the number of carbon atoms, plus two, gives the number of hydrogen atoms.

Let us prove this:

Take methane, CH_4 . There is one carbon— C_n means C.

H_{2n+2} means $2 \times 1 = 2$; $2 + 2 = 4$, H_{2n+2} means H_4 .

So with ethane, C_2H_6 . C_n means C_2 .

H_{2n+2} means $2 \times 2 = 4$; $4 + 2 = 6$, H_{2n+2} means H_6 .

Likewise with propane, C_3H_8 . In this case C_n means C_3 .

H_{2n+2} means $2 \times 3 = 6$; $6 + 2 = 8$, H_{2n+2} means H_8 .

Lastly the hydrocarbon, $\text{C}_{60}\text{H}_{122}$. In this case C_n means C_{60} .

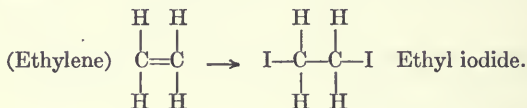
H_{2n+2} means $2 \times 60 = 120$; $120 + 2 = 122$; H_{2n+2} means H_{122} .

In methane, ethane, propane, and the other hydrocarbons, possessing the general formula $\text{C}_n\text{H}_{2n+2}$, the carbon atoms are arranged

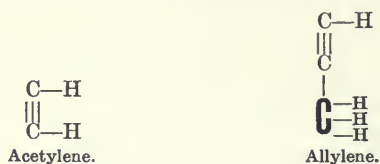
in a chain, that is, one linked after another, like beads on a string. Another variety of chain hydrocarbons are those having the formula C_nH_{2n} , of which ethylene and propylene are the types. These bodies are unsaturated, that is, at least two of the carbon atoms of the compound are linked together by means of double bonds, as outlined in the graphic formula:



These are said to be unsaturated, because the double bonds split whenever opportunity is presented for them to be used for attaching other elements to the carbon atom. Thus, if ethylene is treated with iodine, the double bond is immediately severed and an iodine atom is attached to each bond, making ethyl iodide, as shown in the following graphic formula:



Another series of chain hydrocarbons are the acetylene series, in which adjoining carbon atoms are connected by a triple bond, as shown in acetylene and allylene, graphic formulas of which are given:



Besides these chain series of hydrocarbons, we have a series of hydrocarbons of which benzene, C_6H_6 , is the type, in which the carbon atoms form a ring, as explained on p. 757. This series is called the aromatic series, in contradistinction to the fatty series of chain hydrocarbons just cited.

Radicles are atomic groups, either organic or inorganic, not existing in a free state, but assigned names, and temporarily considered as entities, merely for convenience.

Chemists run across scores of chemical compounds containing the group, say, C_2H_5 (examples: C_2H_5OH , or alcohol; $(C_2H_5)_2O$, or ether, etc.), and as many others containing, say, $COOH$. Neither C_2H_5 nor $COOH$ exist in a free state, but since we meet them so often in compounds, we give them names. Thus, we call C_2H_5 "*ethyl*," simply because it is much easier to say "*ethyl*" than to call the compound "two atoms of carbon and five atoms hydrogen," and for similar reason we call $COOH$ "*carboxyl*." Such atomic groupings are called radicles.

In methane, CH_4 , we have a body in which all the bonds of the

carbon are taken up by hydrogen. In all known bodies the employment of all the bonds of the carbon atoms, in some way or other, is distinctly noticeable, and carbon compounds having free bonds do not exist.

As already mentioned, free atoms do not exist; yet free atoms enter into chemical combinations. Such is the case with what are called radicles, and of these, we may take as example the group CH_3 , which we call methyl.

Note that $\text{C} \begin{smallmatrix} \text{—H} \\ \text{—H} \\ \text{—H} \end{smallmatrix}$ has a free bond, hence it does not exist in a free state. But as it enters into chemical reactions, we may consider it just as we do atoms.

For instance, just as sodium (Na) and chlorine (Cl) join to form sodium chloride (NaCl), so is obtained CH_3Cl , which is called methyl chloride.

Just as the radicle CH_3 is considered as a derivative of CH_4 , so from C_2H_6 is derived the radicle *ethyl*, C_2H_5 , and from C_3H_8 the radicle C_3H_7 , which is called *propyl*.

Another similarity between atoms of elements and radicles.

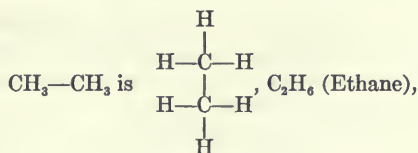
Elements are divided into positive and negative, according as their action resembles metals or non-metals.

The radicles mentioned thus far are all typical of a character somewhat similar to metals, since each (CH_3 , for example) combines with chlorine, just as sodium or iron does. Others there are that act like the negative part of the molecule; such groups as (OH), (COOH), and (CHO) resembling negative elements.

But too much trust must not be placed on this idea of radicles, as the following facts show that they should be considered more as a convenient figure of speech than as definite chemical entities.

Na, the atom of sodium, does not exist. Na_2 , the molecule, does (graphically expressed, Na—Na), the atoms linked "hand in hand."

Methyl, the radicle CH_3 , does not exist; $(\text{CH}_3)_2$ does, but as a body entirely different from the hypothetic methyl, from which it is derived.



which, when brought in contact with chlorine, does not form CH_3Cl (as Na_2 treated with chlorine yields NaCl), but the product is $\text{C}_2\text{H}_5\text{Cl}$.

Another difference is that the atom of an element is unchangeable; methyl, the radicle, can be modified from $(\text{CH}_3)^1$ to $(\text{CH}_2\text{Cl})^1$, $(\text{CHCl}_2)^1$, or $(\text{CCl}_3)^1$, and exactly so with all other radicles.

The Roman numeral outside the brackets in each formula means that the radicles have as many free bonds as the numeral indicates.

Further discussion of radicles is beyond the limits of this book, and in conclusion there will only be added the statement that the name of each radicle is given the termination "*yl*."

The following are the more important radicles:

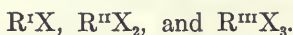
POSITIVE.		NEGATIVE.
Methyl (CH_3) ^I ,	from methane, CH_4 .	Hydroxyl (OH) ^I .
Ethyl (C_2H_5) ^I ,	from ethane, C_2H_6 .	Carboxyl (COOH) ^I .
Propyl (C_3H_7) ^I ,	from propane, C_3H_8 .	Carbonyl (CO) ^{II} .
Butyl (C_4H_9) ^I ,	from butane, C_4H_{10} .	The Aldehyde Radicle, (CHO) ^I .
Amyl (C_5H_{11}) ^I ,	from pentane, C_5H_{12} .	
Phenyl (C_6H_5) ^I ,	from benzene, C_6H_6 .	

In considering the chemistry of the official organic substances, it is found convenient first to discuss the hydrocarbons themselves, and then the vast mass of derivatives, and in this work the order of the consideration of the latter will be based on a convenient standard of the number of carbon atoms they contain. In special books on organic chemistry it is usual to consider the derivatives of the hydrocarbons in groups, based on their structural characteristics; thus, all the fatty alcohols are considered together, likewise the fatty aldehydes and acids, but, from the pharmacist's point of view, it is better to discuss them in a different arrangement; namely, to study all the derivatives of ethane together, and then proceed to propane.

A convenient grouping of the derivatives is as follows:

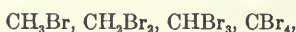
The Halogen Derivatives.—In this case the hydrogen atoms are replaced by the halogen elements, chlorine, bromine, iodine. Thus, from CH_4 we can get CH_3Cl , CH_2Cl_2 , CHCl_3 , and CCl_4 .

The first three of these can be expressed by the formulas:



R standing for the radicle, X for the appropriate halogen element, the Roman numerals above R expressing the number of free bonds the same possesses.

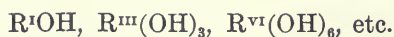
Methane treated with bromine yields respectively—



and such is the case with iodine. Likewise, with ethane, one or even all the halogens can be replaced with chlorine, bromine, or iodine.

Of these halogen derivatives chloroform, bromoform, and iodoform are of pharmaceutic interest.

The Alcohols.—The formulas of these can be expressed by:



Alcohols are substitution products of the hydrocarbons, in which one or more of the hydrogens are replaced by hydroxyl groups (OH). Official examples of this class are:

Ethyl alcohol.....	$\text{C}_2\text{H}_5\text{OH}$.
Glycerin.....	$\text{C}_3\text{H}_5(\text{OH})_3$.
Mannitol.....	$\text{C}_6\text{H}_8(\text{OH})_6$.

These alcohols are divided into primary, secondary, and tertiary alcohols, being classified according to their action under the influence of oxidation.

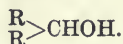
A *primary alcohol* oxidizes to an aldehyde and then to an acid; thus, ethyl alcohol, $\text{CH}_3\text{CH}_2\text{OH}$, on being treated with an oxidizing agent, yields first an aldehyde, CH_3CHO , and finally the acid, CH_3COOH .

A general formula of a primary alcohol shows as distinguishing characteristic the group RCH_2OH , that is, the carbon atom to which the hydroxyl is attached has also attached one radicle and two hydrogen atoms.

A *secondary alcohol*, on oxidation, does not yield an aldehyde and an acid, but yields a substance called a ketone.

Thus, secondary propyl alcohol, $\text{CH}_3\text{CHOHCH}_3$, yields on oxidation the ketone, acetone, CH_3COCH_3 .

The rational formula of a secondary alcohol shows the characteristic grouping—



In other words, connected with the carbon atom to which the hydroxyl is attached are found two radicles and only one hydrogen atom.

A *tertiary alcohol* is one which, on oxidation, yields neither an aldehyde nor a ketone, but breaks up under such treatment. The rational formula of tertiary alcohol can best be expressed as—



in other words, to the carbon atom to which the hydroxyl is attached there are fastened three radicles and no hydrogen atoms at all.

Perhaps the differentiation in the three classes of alcohols can better be understood by a comparison of three such alcohols from the same hydrocarbon and the oxidation derivatives of each.

Thus, primary butyl alcohol, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$, yields on oxidation butyric aldehyde and butyric acid.

Secondary butyl alcohol, $\begin{array}{c} \text{CH}_3\text{CH}_2 \\ \text{CH}_3 \end{array} > \text{CHOH}$, under same treatment, yields methylethylketone, while tertiary butyl alcohol, $\begin{array}{c} \text{CH}_3 \\ \text{CH}_3 \end{array} > \text{COH}$, breaks up into simpler compounds.

Another classification of alcohols is to call them monatomic, diatomic, and triatomic, etc., the Greek prefixes signifying the number of hydroxyls the alcohol possesses; thus glycerin, $\text{C}_3\text{H}_5(\text{OH})_3$, is a triatomic alcohol.

It will be seen that the characteristic radicle of alcohols is hydroxyl (OH). To express the same thing differently, alcohols are hydroxides of the organic radicles, just as the bases (p. 372) are inorganic hydroxides.

Thus, NaOH, sodium hydroxide, has an organic analogue in $\text{C}_2\text{H}_5\text{OH}$, ethyl hydroxide. Similar to $\text{Ca}(\text{OH})_2$, calcium hydroxide, is $\text{C}_2\text{H}_4(\text{OH})_2$, glycol, and as mate to $\text{Fe}(\text{OH})_3$, ferric hydroxide, we have the organic hydroxide, $\text{C}_3\text{H}_5(\text{OH})_3$, glycerin.

All three organic hydroxides cited above are classed among the alcohols. Those alcohols formed by adding hydroxyl to the benzene group of radicles (C_6H_5 , C_6H_4 , etc.) are called *phenols*, of which the type is ordinary phenol, carbolic acid, $\text{C}_6\text{H}_5\text{OH}$.

As alcohols represent organic hydroxides, so the class of *ethers* represent organic oxides. In a preceding chapter (p. 372) it was noted that sodium hydroxide, NaOH, was evolved from sodium oxide, Na_2O , by adding water thereto. Likewise, by taking water away from sodium hydroxide, sodium oxide is produced. In a somewhat analogous way, by removing water from ethyl hydroxide (alcohol), ethyl oxide (ether) is formed.

oxidizing agents, loses the two hydrogen atoms, becoming CH_3COCH_3 , the official acetone.

Acids.—The true organic acids are the oxidation products of primary alcohol or aldehydes, and their general formula is RCOOH , the group COOH being called *carboxyl*, as mentioned on p. 630.

As alcohols are monatomic, diatomic, and triatomic, according to the number of hydroxyls, so organic acids are monobasic, dibasic, and tribasic, according to the number of carboxyls they contain. Thus, acetic acid is an official example of the monobasic organic acid, while succinic acid, $\text{C}_2\text{H}_4(\text{COOH})_2$, is an illustration of a dibasic acid.

Acids with alcohol radicles, that is, containing carboxyl and hydroxyl, are called *oxyacids*, and an official illustration is furnished in tartaric acid, which is a dibasic, diatomic oxyacid, as shown by the graphic formula. (See p. 685.)

In true acids only those hydrogen atoms in carboxyl groups are replaceable by a metal. This is why carboic acid ($\text{C}_6\text{H}_5\text{OH}$) is not a true acid, but a phenol (p. 758).

Amines can be expressed by the general formulas—



They can also be considered as ammonia in which one, two, or three hydrogens have been replaced by radicles. Thus we can obtain methylamine, CH_3NH_2 , dimethylamine, $(\text{CH}_3)_2\text{NH}$, and trimethylamine, $(\text{CH}_3)_3\text{N}$.

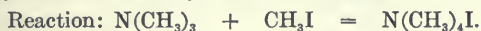
The manufacture of these products is so simple and clear that it serves admirably for explaining their exact composition. Thus ammonia treated with methyl iodide gives the hydriodic acid salt of methylamine, and when the acid is removed by treating with potassa, methylamine remains.



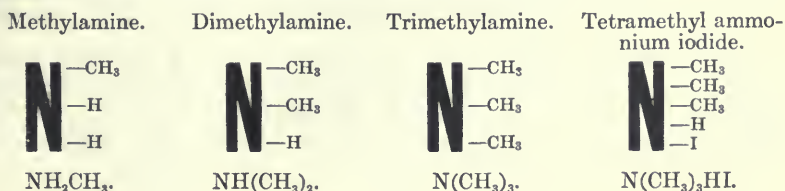
Methylamine, being treated with methyl iodide, will yield the hydriodic acid salt of dimethylamine, and this by treatment with potassa yields the free dimethylamine.



Dimethylamine, being treated with methyl iodide, yields the hydriodic acid salt of trimethylamine, and the free base is obtained by treatment with potassa as above. Lastly, if trimethylamine is treated with methyl iodide, we get a very interesting body called tetramethylammonium iodide.



In the latter case the nitrogen shows the valence v, exactly as it does in the ammonium compounds. The similarity of these four bodies, the amines and the ammonium compounds respectively, is best shown by comparison of the four graphic formulas:



It might be added that for methyl can be substituted ethyl, propyl, or any other monovalent radicle. All the amines are quite similar to ammonia; the mono-amines have ammoniacal odor and all are decidedly basic.

The amines of the fatty series are of but little importance, but several of the aromatic series are of the utmost value; for example, aniline (p. 767).

CHAPTER XXXVI

HYDROCARBONS AND METHANE DERIVATIVES

THE FATTY SERIES OF HYDROCARBONS

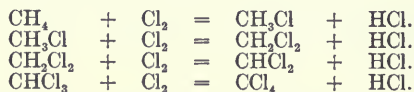
THIS series of hydrocarbons, having the general formula C_nH_{2n+2} , are sometimes called the *aliphatic series*. As mentioned in the preceding chapter, representatives of these series are the homologues, methane, ethane, butane, pentane, hexane, etc., all similar and manufactured one from the other. Substances possessing the same general formula, but differing in relative number of atoms, are called *homologues*.

Manufacture.—That the members of this class can be made one from the other has already been mentioned; thus, methane, CH_4 , can be converted into methyl iodide, (CH_3I) , and methyl iodide, on treatment with metallic sodium, yields ethane by the following reaction:



This is the usual way of making such hydrocarbons, although several other methods have been devised.

Chemical Behavior.—These hydrocarbons are saturated bodies; that is, they will absorb no more hydrogen nor will they combine with halogens except through the loss of hydrogen atoms. The halogen derivatives are formed as follows:

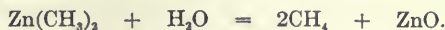


Physical Properties.—The first of these series of hydrocarbons are gases. Hydrocarbons from C_6H_{14} are liquids, the first being quite volatile, the higher ones, that is, from $C_{14}H_{30}$, thick and stable liquids, while the higher hydrocarbons of the series, say from $C_{20}H_{42}$, are solid.

Considering the hydrocarbons as individuals, the first of the series, *methane*, CH_4 , is commonly called *marsh-gas*, because formed by decaying vegetable matter, and thus it can be easily collected from the surfaces of some marshes. It is the chief constituent of natural gas. Methane is usually made by heating in a retort sodium acetate with sodium hydroxide, as shown in the following equation:



It can also be prepared spontaneously by immersing zinc methyl in water, as shown in the following equation:



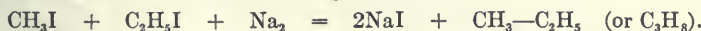
The zinc methyl used is made by heating together methyl iodide and zinc and distilling over the zinc methyl in the presence of absolutely dry hydrogen, the reaction being:



Ethane, C_2H_6 , can be made as cited on p. 634, by treatment of methyl iodide with metallic sodium. It is usually made, however, by treating sodium propionate with sodium hydroxide in a manner similar to the method given for making methane from sodium acetate, the reaction being as follows:

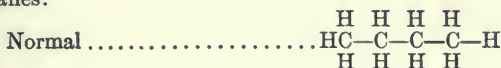


Propane, C_3H_8 , is made by treating a mixture of methyl iodide and ethyl iodide with metallic sodium, the reaction being as follows:

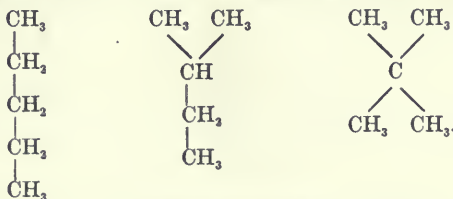


Butane, C_4H_{10} , is interesting, inasmuch as two substances of this formula— C_4H_{10} —are known. Already we have learned that where an element is found in three or four different forms, such forms are said to be allotropic (p. 397). On the other hand, where several compounds differ physically, but have the same chemical formula, they are called *isomers*, and in organic chemistry a vast number of such isomers are found, the two types of butane being the first example we meet. The two types of butane can best be described by submitting the graphic formula, and it will be found, on careful calculation, that these are the only two possible combinations of the formula C_4H_{10} :

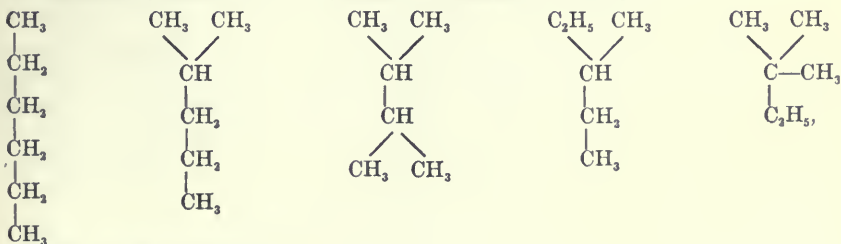
The two butanes:



Pentane, C_5H_{12} , can occur in three different forms, namely:



Hexane, C_6H_{14} , exists in five possible combinations, namely:



and all of these are known.

It will be seen from the above that the number of isomeres increases in proportion to the number of carbon atoms which the compound possesses, and in bodies containing a large number of carbon atoms the possible number of isomeres is enormous.

Of the hydrocarbons beyond hexane, such as C_7H_{14} and C_8H_{18} , nothing need be said beyond the fact that the formula of each can be worked out from the general formula C_nH_{2n+2} , as explained on p. 627.

The hydrocarbons above cited are of interest pharmaceutically because they are constituents of natural gas and of the petroleum products, and these we will now discuss.

Natural gas is chiefly methane combined with small quantities of ethane and oxygen. It is found in pockets beneath the surface of the earth, from which it is extracted by drilling, as in artesian wells, until the pocket is reached, when the gas rushes out and is piped to places where it is desired to be used. The city of Cleveland is furnished with natural gas brought from West Virginia, 120 miles away.

Petroleum is a thick, greasy liquid, obtained from pockets beneath the earth by drilling, as in the case of natural gas—in fact, the two are usually associated. The chief localities in which petroleum is obtained are Pennsylvania and the Central Western States (Ohio, Indiana, West Virginia), Texas, and in the neighborhood of Baku, South Russia.

Petroleum was first drilled for in Venango County, Pennsylvania, in 1859, although the oil which oozed from the earth was known by the Indians two hundred and fifty years ago. The Russian fields have been worked commercially only since the Pennsylvania fields have been operated, while the Texas fields were opened in 1900.

Petroleum is supposed to be the result of the decomposition of decaying fishy matter, the geological explanation of the limited areas where found being that in each case the pockets represent some sea that disappeared, these cataclysms of nature leaving the fishy denizens to be engulfed beneath masses of rock. It is interesting to note that the destructive distillation of products other than fish yield substances similar to petroleum. Thus linseed, on being submitted to such distillation, gives a petroleum-like distillate.

Of the petroleum, that of Pennsylvania and adjoining states is the best, the Russian containing sulphur, which must be removed from the distillate before it can be used for fuel or light, while the Texas petroleum contains a lesser amount of the lower boiling products and contains 1.4 to 1.8 per cent. sulphur. As mentioned above, the process of extracting petroleum from the earth is very simple, consisting merely of driving an artesian well, and when the bed-rock is reached, exploding the rocky layer with nitroglycerin. The depth of this rocky layer varies, some wells being 2600 feet deep.

When the free petroleum rushes through the tube to the earth's surface, it is with a pressure, more or less great, depending on the amount of gas mixed with petroleum. The Russian petroleum is said to be much more gaseous than the American, and therefore sprouts to a greater height, some rising 300 feet at first.

The Texas petroleum was originally under great pressure, the famous Spindle Top "Gusher" of Beaumont spouting originally to 150 feet.

At first the gas pressure is sufficient to cause a large flow of petroleum; in fact, in some cases the difficulty is properly to control the flow. After flowing a while, however, the pressure diminishes, until finally recourse must be had to pumps.

Petroleum, as it emerges from the earth, is a dark, greasy liquid of disagreeable odor. This forms "*stone oil*," which was formerly in considerable esteem medicinally as a liniment. Petroleum is pumped into tanks and purified from extraneous organic matter by treatment with sulphuric acid. The petroleum layer is then distilled, yielding:

1. Gases, such as methane, CH_4 , and ethane, C_2H_6 .
2. Rhigolene, chiefly C_3H_{12} , boiling-point, $10^\circ C$.
3. Gasoline (benzin), chiefly C_5H_{12} and C_6H_{14} , boiling-point, 50° – $60^\circ C$.

4. Insurance oil, coal oil, etc., C_7H_{16} , C_8H_{18} , etc., boiling-point, 60° – 100° C.
5. Liquid petrolatum, U. S. P. } { Residue left after distillation of the
6. Petrolatum, U. S. P. } { more volatile portion. They are
7. White petrolatum, U. S. P. } { separated from each other by
8. Paraffin. } { expression and distillation in vacuo.

Rhigolene has been suggested (on account of its great volatility) as a local anesthetic, it freezing the surface on which it is sprayed. However, ethyl chloride is said to be better for this purpose.

BENZINUM. Petroleum Benzin.

A distillate from American petroleum consisting of hydrocarbons, chiefly of the marsh-gas series [C_5H_{12} , C_6H_{14} , and homologous compounds]. It should be carefully kept in well-stoppered bottles or tin cans, in a cool place, remote from lights or fire.

A transparent, colorless, diffusive liquid, of a strong, characteristic odor, slightly resembling that of petroleum, but much less disagreeable, and having a neutral reaction. Petroleum benzin is highly inflammable, and its vapor, when mixed with air and ignited, explodes violently.

Specific gravity: 0.638 to 0.660 at 25° C. (77° F.).

Insoluble in water; soluble in about 6 parts of alcohol and readily soluble in ether, chloroform, benzene, volatile oils, and fixed oils, with the exception of castor oil.

Boiling-point: 45° to 60° C. (113° to 140° F.).

If 5 drops of Petroleum Benzin be added to a mixture of 40 drops of sulphuric acid and 10 drops of nitric acid, in a test-tube, the liquid warmed for about ten minutes, and then set aside for half an hour, on diluting it, in a shallow dish, with water, it should not evolve the bitter-almond-like odor of nitrobenzene (difference from, and absence of, *benzene*).

The synonym for this important substance is *petroleum ether*, and under this name should be invariably ordered, for the commercial benzin or gasoline consists chiefly of higher boiling hydrocarbons of petroleum.

The commercial samples examined by the writer, instead of distilling over at 50° C., as directed by U. S. P., did not begin to boil until 85° C. was reached, while practically all distilled over between 95° and 100° C.

Benzin is directed by the pharmacopœia as a solvent for India-rubber (when mixed with equal quantities of carbon disulphide), and is thus used in making mustard paper. Its value as a remover of grease-spots is well known.

BENZINUM PURIFICATUM. Purified Petroleum Benzin.

Recipe.—Potassium Permanganate, ten grammes..... 10 Gm.
Sodium Hydroxide, two grammes..... 2 Gm.
Sulphuric Acid, sixty cubic centimeters..... 60 Cc.
Petroleum Benzin, one thousand cubic centimeters. 1000 Cc.
Water, a sufficient quantity.

Add the Acid to five hundred and fifty cubic centimeters of Water, and when the mixture has become cold, pour it into a bottle having the capacity of about two liters. Add eight grammes of Potassium Permanganate and agitate until it is dissolved, then add the Petroleum Benzin, in four portions, shaking the liquid after each addition. Allow the liquids to remain in contact for twenty-four hours, shaking the bottle at frequent intervals; then decant the Petroleum Benzin into another bottle of the same

capacity, and having dissolved *two grammes* of Potassium Permanganate in *two hundred and forty cubic centimeters* of Water, in which the Sodium Hydroxide has previously been dissolved, mix the liquids and agitate the mixture frequently during several hours, then decant, repeat the washing with Water, and again decant the Purified Petroleum Benzin.

On evaporating 10 Cc. of Purified Petroleum Benzin from a piece of clean filtering paper no greasy stain should remain, and the odor should not be disagreeable or notably sulphuretted; no residue should be left upon evaporating Purified Petroleum Benzin from a warmed dish (absence of *heavy hydrocarbons*).

When it is boiled for a few minutes with one-fourth its volume of spirit of ammonia and a few drops of silver nitrate T.S., the liquid should not turn brown (absence of *pyrogenous products* and *sulphur compounds*).

Purified Petroleum Benzin should have an ethereal or faint petroleum-like odor, and should respond to the tests given under *Benzinum*.

Remarks.—This represents the crude petroleum ether free from other matter by oxidation with permanganate. It is directed for use by the present pharmacopœia as a solvent for the caoutchouc-like constituent of lactucarium in the manufacture of tincture of lactucarium. Also for the removal of the odorous principles of opium in making tincture of deodorized opium.

Gasoline in commerce is a term used synonymously with benzin, its chief use being for burning in the so-called vapor stoves. The various illuminating oils, such as *insurance oil*, *coal-oil*, *kerosene*, *headlight oil*, and *astral oil*, are petroleum products distilling over between 60° and 150° C. The higher the boiling-point, the safer is such an oil; as a matter of fact, the use of the lower boiling oils, such as benzin and gasoline, are dangerous, especially in wick lamps. As the last two are cheaper than the higher boiling oils, because more abundant, they are used to adulterate the higher boiling oils. To detect such adulteration has been devised the so-called "*flash test*," which consists in heating the sample in an appropriate apparatus and noting at what temperature the sample gives off inflammable vapors. Thus kerosene oil has a flash test of 45° C.; that is, its vapors should not ignite until the oil is heated to that temperature.

On the other hand the flash test of astral oil is about 150° C.

The first distillate of these bodies is always impure, and must be purified by treatment with sulphuric acid and redistillation.

The pharmaceutical products, *liquid petrolatum*, *soft petrolatum*, and *hard petrolatum*, are obtained from the residue after distillation. This residue is distilled in vacuo, and according to its consistence forms hard or soft petrolatum, the latter having been introduced by Robert Chesebrough in 1871 under the name of "*vaseline*." If, on the other hand, the purified residue is chilled and then expressed, the limpid oil flows out, forming *liquid petrolatum*, U. S. P., while the residue is the commercial *paraffin*. The purification of the product in each case is accomplished by filtration through animal charcoal.

PETROLATUM LIQUIDUM. Liquid Petrolatum.

A mixture of hydrocarbons, chiefly of the methane series, obtained by distilling off most of the lighter and more volatile portions from petroleum, and purifying the liquid residue.

A colorless or very slightly yellowish, oily, transparent liquid, without odor or taste, but giving off, when heated, a faint odor of petroleum.

Specific gravity: about 0.870 to 0.940 at 25° C. (77° F.).

It is insoluble in water; scarcely soluble in cold or hot alcohol, or in cold absolute alcohol, but soluble in boiling absolute alcohol, and readily soluble in ether, chloroform, carbon disulphide, oil of turpentine, petroleum benzin, benzene, and fixed or volatile oils.

When heated on platinum foil, Liquid Petrolatum is completely volatilized without emitting acrid vapors.

If a test-tube be half filled with Liquid Petrolatum and a piece of moistened blue litmus-paper be introduced, upon shaking the liquid vigorously the paper should not be reddened (absence of *acid impurities*).

In other respects Liquid Petrolatum has the characteristics of, and should respond to, the tests given under *Petrolatum*.

Liquid petrolatum has now considerable use as the basis of atomizing fluids. The pharmacopœia directs colorless or a very slightly yellowish, oily, transparent liquid, but it is good pharmacy invariably to dispense in prescriptions the colorless product, such as "*liquid albolene*." The solvent powers of liquid petrolatum are limited to the phenols, such as menthol, thymol, and to a few of the organic chemicals.

PETROLATUM. Petrolatum.

[PETROLATUM MOLLE, PETROLATUM SPISSUM, PHARM. 1890]

A mixture of hydrocarbons, chiefly of the methane series, obtained by distilling off the lighter and more volatile portions from petroleum, and purifying the residue.

An unctuous mass, of about the consistence of an ointment, varying in color from yellowish to light amber, having not more than a slight fluorescence, even after being melted, transparent in thin layers, completely amorphous; without odor or taste, but giving off, when heated, a faint petroleum-like odor.

If a portion of Petrolatum be liquefied and brought to a temperature of 60° C. (140° F.), it should have a specific gravity of from 0.820 to 0.850.

Petrolatum is insoluble in water; scarcely soluble in cold or hot alcohol, or in cold, absolute alcohol, but soluble in boiling absolute alcohol, and readily soluble in ether, chloroform, carbon disulphide, oil of turpentine, petroleum benzin, benzene, and fixed or volatile oils.

The melting-point of Petrolatum ranges between 45° and 48° C. (113° and 118.4° F.). If heated on platinum foil to a still higher temperature, Petrolatum should be completely volatilized without emitting any acrid odor.

If melted Petrolatum be well shaken with water, the latter should not redden blue litmus-paper.

If 10 Gm. of Petrolatum be digested at 100° C. (212° F.) for half an hour with 10 Gm. of sodium hydroxide and 50 Cc. of water, the aqueous layer separated and supersaturated with sulphuric acid, no oily or solid substance should separate (absence of *fixed oils* or *fats of animal or vegetable origin* and of *rosin*).

If 2 volumes of concentrated sulphuric acid be added to 1 volume of melted Petrolatum in a test-tube placed in hot water, and the contents occasionally agitated during fifteen minutes, the acid should not acquire a deeper tint than brown nor lose its transparency (limit of *readily carbonizable organic impurities*).

Remarks.—Under the heading Petrolatum the present pharmacopœia includes the hard and soft petrolatum of the pharmacopœia of 1890. *Soft petrolatum* is the well-known form of *petroleum jelly*, appearing in commerce under the trade names of *vaseline*, *cosmoline*, *sazolene*, etc.

PETROLATUM ALBUM. White Petrolatum.

A colorless mixture of hydrocarbons, chiefly of the methane series, obtained by distilling off the lighter and more volatile portions from petroleum, and purifying the residue.

A white, unctuous mass of about the consistence of an ointment, transparent in thin layers, completely amorphous; without odor or taste. In other respects White Petrolatum has the characteristics of, and should respond to, the tests given under *Petrolatum*.

Remarks.—While the pharmacopœia of 1890 permitted the dispensing, under the title "*petrolatum*," either the white or the pale

yellow variety, the present pharmacopœia distinguishes between the white and the yellow as above described.

White petrolatum represents a special form of solid petrolatum obtained from Russian petroleum. It was originally introduced in this country under the name of *albolene*. Incidentally, it might be mentioned that the colorless liquid petrolatum is technically termed "Russian White Oil."

A cheap form of white petrolatum, made by dissolving paraffin in liquid petrolatum in varying quantities, has been put on the market. This, however, should never be used, as on standing it invariably becomes lumpy, due to separation of the paraffin.

These petroleum jellies are used as ointment bases, possessing over lard and other fats the great advantage of not turning rancid. Because of their greasy character, these jellies are usually grouped among the animal and vegetable fats, but from these the petroleum products differ entirely, the latter being the glycerin derivatives of oleic, stearic, and palmitic acids; their rancidity is due to their decomposition, with liberation of the fatty acids just named. This is, of course, impossible with petrolatum, which is a mixture of the hydrocarbons, and, like all hydrocarbons, are not easily decomposed, even by chemicals. In fact, the halogens are about the only substances which will combine directly with the hydrocarbons.

This chemical stability is of great advantage in so far as the absence of rancidity is concerned, but, on the other hand, in making ointments, its stability is in some cases disadvantageous. Thus, physicians sometimes prescribe alkalis with fats, with the intention of securing a form of soap. If, instead of the animal or vegetable fat, the physician prescribes a petrolatum product, the same results are not obtained and an unsightly mixture occurs. An example of this is found in prescriptions (p. 1082).

Another drawback to the use of petroleum jelly as an ointment base is the fact that it is by no means as easily absorbed by the skin as are the animal or vegetable fats.

PARAFFINUM. Paraffin.

A mixture of solid hydrocarbons, chiefly of the methane series; usually obtained by chilling and pressing the distillates from petroleum having high boiling-points, and purifying the solid press cake so obtained.

A colorless, more or less translucent mass, crystalline when separating from solution; without odor or taste and slightly greasy to the touch.

Specific gravity: from 0.890 to 0.905 at 25° C. (77° F.).

Insoluble in water or alcohol; slightly soluble in absolute alcohol; readily soluble in ether, petroleum benzin, benzene, carbon disulphide, volatile oils, and in warm fixed oils.

Its alcoholic solution should not redden moistened blue litmus-paper.

When heated it melts at from 51.6° to 57.2° C. (125° to 135° F.), and on stronger heating ignites, burning with a luminous flame and depositing carbon, but leaving no permanent residue.

If 0.5 Gm. of Paraffin be heated in a dry test-tube with an equal weight of sulphur, the mixture will become black from the separated carbon, with the evolution of hydrogen sulphide gas.

Paraffin is not acted upon or colored by concentrated sulphuric acid or nitric acid in the cold.

If 0.5 Gm. of Paraffin be heated and 0.1 Gm. of powdered fuchsin added to the fused mass, the latter should not assume a pink or red color (absence of *stearic acid*).

Remarks.—Paraffin consists of the hydrocarbons from $C_{24}H_{50}$ to $C_{27}H_{56}$, or possibly four times the formula just given. It has of late come into extensive use for such purposes as making wax-paper, candles, and for the polishing of floors and other wood.

Like the liquid and soft petrolatums, it is practically impervious to ordinary chemicals. It can even be boiled with concentrated sulphuric acid without any change in its properties, this test being employed in examining beeswax to see if adulterated with paraffin (p. 740).

Cerasin is a purified form of *ozokerite*, a paraffin-like substance mined in Galicia.

The compounds of methane of pharmaceutic interest are:

Methyl chloride.....	CH_3Cl .
Chloroform, U. S. P.....	$CHCl_3$.
Bromoform, U. S. P.....	$CHBr_3$.
Iodoform, U. S. P.....	CHI_3 .
Methyl alcohol.....	CH_3OH .
Formic acid.....	$HCOOH$.
Formaldehyde.....	$HCHO$.
Solution of formaldehyde, U. S. P., contains 37 per cent....	$HCHO$.
Methyl salicylate, U. S. P.....	$CH_3C_7H_5O_3$.

Methyl Chloride (CH_3Cl).—This is made by treating methyl alcohol with hydrochloric acid in the presence of zinc chloride (Grove's method), the zinc chloride being used as a dehydrator—that is, to remove the water. It is now produced commercially from *schlemp*, or the refuse in the beet sugar manufacture. Methyl chloride is a colorless gas, and is put upon the market in liquid form which boils at $22^\circ C$., made by compressing the gas, and is used as a solvent in perfumery and in synthesis of dye-stuffs. Its medicinal use is for the purpose of producing local anesthesia, by directing it in a stream upon the part desired to become benumbed, as was the case in rhigolene.

CHLOROFORMUM. Chloroform.

A liquid consisting of 99 to 99.4 per cent., by weight, of absolute Chloroform [$CHCl_3 = 118.45$] and 0.6 to 1 per cent. of alcohol. It should be kept in dark amber-colored, glass-stoppered bottles, in a cool and dark place.

A heavy, clear, colorless, mobile and diffusible liquid, of a characteristic, ethereal odor, and a burning, sweet taste.

Specific gravity: not below 1.476 at $25^\circ C$. ($77^\circ F$.).

Soluble in about 200 times its volume of cold water, and, in all proportions, in alcohol, ether, benzene, petroleum benzin, and the fixed and volatile oils.

Chloroform is volatile even at a low temperature and boils at 60° to $61^\circ C$. (140° to $141.8^\circ F$.). It is not inflammable, but its heated vapor burns with a green flame.

If 10 Cc. of Chloroform be poured upon a piece of clean, odorless filter-paper laid flat upon a warmed glass plate, and the plate be rocked from side to side until the liquid is all evaporated, no foreign odor should become perceptible as the last portions disappear from the paper, and the paper should be left odorless.

If 10 Cc. of Chloroform be well shaken with 20 Cc. of distilled water, and the liquid be allowed to separate completely, the water should be neutral to litmus-paper, and should not be affected by silver nitrate T.S. (absence of *chlorides*) nor colored by potassium iodide T.S. (absence of *free chlorine*).

If 40 Cc. of chloroform be shaken with 4 Cc. of colorless, concentrated sulphuric acid in a 50 Cc. glass-stoppered cylinder during twenty minutes, and the liquids be then allowed to separate completely, so that both are transparent, the chloroform should remain colorless, and the acid should appear colorless, or very nearly colorless, when seen in a stratum of not less than 15 Mm. in thickness (absence of *impurities decomposable by sulphuric acid*).

If 2 Cc. of the sulphuric acid, separated from the Chloroform, be diluted with 5 Cc. of distilled water, the liquid should be colorless and clear, and, while hot from the mixing, should be odorless, or give but a faint vinous or ethereal odor (absence of *odorous decomposition products*). When further diluted with 10 Cc. of distilled

water, it should remain clear and should not be affected by silver nitrate T.S. (absence of *chlorinated decomposition compounds*).

Remarks.—Chloroform was discovered by Dr. Guthrie, of Jewettsville, N. Y., in 1831, and was considered merely as an interesting chemical specimen until after the discovery of anesthesia by Long in 1842, when it was discovered that chloroform could be used for that purpose as well as could ether, and since that time it has been of untold benefit in alleviating suffering attendant upon surgical operations.

Chloroform is made by treating alcohol with chlorinated lime. Chlorinated lime is, as learned on p. 392, a loose combination of chlorine and the alkali, lime; chlorine acting on alcohol gives chloral, as will be learned on p. 668, and chloral in contact with an alkali gives chloroform.

As this method of manufacture is so similar to that of chloral, full explanation of the process had best be left until we consider that chemical. The same applies to the manufacture of a very pure chloroform by treatment of chloral with an alkali.

Of recent years a large amount of chloroform has been produced by the so-called acetone process, patented by Michaelis, in which acetone is treated with chlorinated lime in the same way as in the alcohol process, the advantage of the acetone process over the alcohol process being that the yield is very good, about 200 per cent., and the product much purer than that yielded by the alcohol process. Equation showing the acetone process is as follows:



The chloroform for anesthesia is always purified by treatment with sulphuric acid and redistillation.

Another method of obtaining absolutely pure chloroform is that of Pictet. Chloroform solidifies at 80° C., whereas all the other impurities are solidified at 70° C. Therefore, the mixture is subjected to a temperature of 70° C., and the still liquid chloroform poured off from the frozen impurities.

A very delicate test for chloroform is the so-called *isonitrile reaction*, this consisting of treating the chloroformic liquid with an amido-compound (for example, aniline) in the presence of an alkali, when the disgusting odor of the poisonous gas, phenyl (or some other) isonitrile ($\text{C}_6\text{H}_5\text{NC}$) is evolved. This test is interesting as being equally valuable for identifying the amines, any of which can be employed in place of aniline. The equation showing the reaction is as follows:

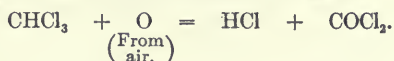


It will be noticed that the pharmacopœia permits the presence of 1 per cent. of alcohol, this being added to preserve same, it being found that pure chloroform decomposes. The fact that chloroform does undergo decomposition renders it necessary for the pharmacist to assure himself that the chloroform he dispenses for anesthesia is strictly pharmacopœial, for should a death result during anesthesia, one of the first things done by the physician is to investigate the purity of the chloroform, and if found impure, it will prove very

disastrous to the reputation of the pharmacist dispensing same. Hence every sample of chloroform should be carefully tested according to the pharmacopœia for chlorinated products and for empyreuma.

A very simple and satisfactory test for empyreuma is the sulphuric acid test: when a layer of chloroform is poured upon pure sulphuric acid, no coloration should take place at point of contact. A simple test for acidity in chloroform is the fact that pure chloroform does not attack a cork of a container. If, therefore, the cork softens or becomes yellow, it is a sure indication of the presence of acid. Of course, the accurate test for acidity is by the use of litmus-paper.

Chloroform allowed to vaporize in the presence of artificial light or heat becomes oxidized, the reaction being:



The latter body, COCl_2 (called *phosgene*), has a very disagreeable odor and is poisonous, hence it is a dictum in surgery that a patient should never be anesthetized by any artificial light except electric.

Besides this employment, as an anesthetic by inhalation, chloroform is used internally as a sedative and externally as a rubefacient.

Dose.—0.3 Cc. (5 minims).

BROMOFORMUM. Bromoform.

A liquid consisting of 99 per cent., by weight, of absolute Bromoform [CHBr_3 = 250.99] and 1 per cent. of absolute alcohol. It should be kept in dark amber-colored, glass-stoppered bottles, in a cool place, protected from light.

A heavy, transparent, colorless, mobile liquid, with an ethereal odor, and a penetrating, sweet taste resembling that of chloroform.

Specific gravity: 2.808 at 25° C. (77° F.).

Very slightly soluble in water, but soluble in all proportions in alcohol, ether, benzene, petroleum benzin, and in the fixed and volatile oils.

Bromoform is slightly volatile at ordinary temperatures, boils at 148° C. (298.4° F.), and solidifies at 6° C. (42.8° F.).

It is not inflammable, but when vaporized by the application of heat its vapor may be burned.

If 10 Cc. of Bromoform be evaporated in a dish over a naked flame, no solid residue should remain.

If 10 Cc. of Bromoform be well shaken with 10 Cc. of distilled water, and the liquids, upon standing, be allowed to separate completely, the water removed from the layer of Bromoform should be neutral to blue litmus-paper (absence of *free acid*), and a portion should not produce a turbidity when treated with silver nitrate T.S. (absence of *bromides* and *brominated compounds*), and another portion treated with potassium iodide T.S. should not be tinted blue upon the addition of starch T.S. (absence of *free bromine*).

If 10 Cc. of Bromoform be well shaken with 10 Cc. of distilled water, and the liquids, upon standing, be allowed to separate completely, the water removed from the Bromoform, treated with an excess of ammonia water, and then with a solution of iodine and ammonium iodide until the black precipitate of nitrogen iodide, which may sometimes form, slowly disappears, it should not become milky in appearance, due to the separation of iodoform, which may be recognized by its odor (absence of *acetone*).

Remarks.—This new official is made by treating alcohol with bromine in the presence of an alkali and distilling the bromoform resulting from the reaction. Bromoform has been recommended as a sedative, particularly in whooping-cough.

Dose.—0.2 Cc. (3 minims).

IODOFORMUM. Iodoform.



Triiodomethane, usually obtained by the action of iodine upon alcohol, in the presence of an alkali or alkali carbonate. Iodoform should be kept in well-stoppered bottles, in a cool and dark place.

A fine, lemon-yellow powder or lustrous crystals of the hexagonal system, having a peculiar, very penetrating and persistent odor, and an unpleasant, slightly sweetish and iodine-like taste.

Soluble in 9391 parts of water, to which it imparts its odor and taste, in 46.7 parts of alcohol, and in 5.2 parts of ether at 25° C. (77° F.); soluble in about 12 parts of boiling alcohol; soluble in chloroform and fixed and volatile oils; slightly soluble in petroleum benzin.

Digest in a porcelain dish 0.1 Gm. of Iodoform with 5 Cc. of an alcoholic solution of potassium hydroxide (1 in 20) until it is dissolved, evaporate to dryness on a water-bath, dissolve the residue in 5 Cc. of distilled water, add 2 Cc. of Chloroform and an excess of nitric acid, and shake the mixture; the chloroform will assume an intense violet color.

Its solutions in neutral solvents are neutral to litmus-paper.

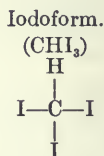
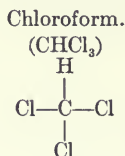
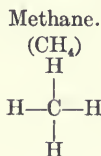
Iodoform is slightly volatile, even at ordinary temperatures, and in boiling water distils slowly with the vapor of water. At about 115° C. (239° F.) it melts to a brown liquid, and at a higher temperature emits vapors of iodine, leaving behind a carbonaceous mass, which, upon full combustion, should leave not more than 0.1 per cent. of residue (limit of *fixed impurities*).

On being dried over sulphuric acid, the loss in weight should not exceed 1 per cent.

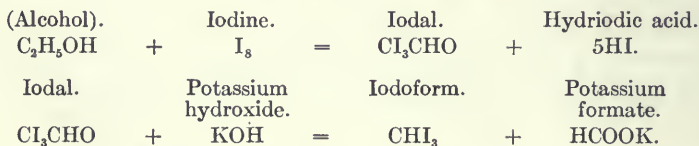
If 2 Gm. of Iodoform be thoroughly shaken with 10 Cc. of water, the filtrate should be colorless and free from bitter taste (absence of *soluble yellow coloring matters*, *picric acid*, etc.); it should not affect the color of litmus-paper (absence of *free acids*), nor should it be rendered more than faintly opalescent by silver nitrate T.S. (absence of *soluble iodides*).

Remarks.—Iodoform is a product chemically very similar to chloroform.

Chloroform is trichlormethane; iodoform is tri-iodomethane. A graphic comparison may be useful:



Iodoform is made by treating alcohol with (the) iodine in the presence of an alkali, the reaction being as follows:



It will be seen in the above reaction that only three of the eight atoms of iodine employed are converted into iodoform, the other five forming hydriodic acid. In view of the high cost of iodine such waste cannot be permitted, and, therefore, chlorine is passed through the reacting liquid, with the formation of HCl and liberation of iodine,

the five atoms of which are in time converted into iodoform. In practice, potassium bicarbonate is the alkali employed in the above reaction.

The manufacture of iodoform, by the way, affords an excellent test for alcohol, even when present in minute proportions. To the alcoholic liquid is added a grain or so of iodine and a few drops of solution of potassa, and the mixture is gently warmed. If there settles out a yellow precipitate of the characteristic iodoform odor, the liquid contains alcohol.

Iodoform, which was discovered by Serullas in 1822, is one of the most valuable antiseptics, the sole objection to its use being its very disagreeable odor. Many efforts are noted in pharmaceutical journals for masking this odor, but none have proved satisfactory, about the most successful masker being coumarin.

The antiseptic properties of iodoform being due to iodine, several odorless iodine preparations have been put on the market as substitutes for iodoform, but none have met with very striking success. The most popular of these iodoform substitutes are *iodol* (p. 753) and *aristol* (p. 801), and neither of these can approach iodoform in actual percentage of iodine. Iodoform contains 96 per cent. of iodine, as an estimation of the molecular weight will show, while iodol contains 84 per cent. and aristol contains 46 per cent.

Dose.—250 milligrammes (4 grains).

Methyl alcohol (CH_3OH) was obtained by Robert Boyle in 1661 as a product in the destructive distillation of wood. By reason of its source its common name is *wood alcohol*, while a purer form of wood alcohol has been put on the market under the trade name of *Columbian spirits*.

Of theoretic interest is the manufacture of wood alcohol from methyl salicylate or oil of wintergreen, this product being treated with sodium hydroxide, yielding sodium salicylate and methyl hydroxide or wood alcohol (p. 785).

The commercial manufacture, however, is by the destructive distillation of wood. Wood cut into billets, placed in iron retorts, and heated, yields, besides gases, a tarry liquid called wood vinegar, or crude *pyroligneous acid*, which consists of a mixture of methyl alcohol, acetone, acetic acid, and a large number of other products.

In the distillation, temperature beyond 218°C . is unnecessary, the yield of distillate being no greater at the higher temperature, while at the temperature just stated the product contains much less *empyreuma*, the last word meaning the tarry odor possessed by organic bodies which have been subjected to destructive distillation.

From the crude distillate acetic acid is first removed by treatment with lime or chalk, forming calcium acetate. The product is then treated with calcium chloride, which causes the wood alcohol to crystallize out in a body, $\text{CaCl}_2\cdot\text{CH}_3\text{OH}$. From these crystals wood alcohol is obtained by distillation, while acetone is gotten from the filtrate by special treatment, to be mentioned later.

Wood alcohol is a valuable solvent, and has been suggested for use in external preparations and for such preparations as extracts, from which the solvent is eventually evaporated.

Chemically pure wood alcohol is said to be non-toxic, but ordinary commercial wood alcohol should never be used as a solvent for internal preparations, for recently, in various sections of the country, the employment of essence of ginger from wood alcohol has resulted in blindness of the unfortunate tipplers.

In a suit brought by one of the victims it was claimed that the disastrous effect was due not to the methyl alcohol, but to the furfurol it contains.

By reason of the disastrous effects of the administration of wood alcohol the use of this chemical in preparations for internal use is prohibited in many states and by the National Pure Food Law, and the present pharmacopœia gives specific warning of its presence in grain alcohol, prescribing a test for its detection.

Methyl ether, $(\text{CH}_3)_2\text{O}$, is unofficial, and is mentioned only as the first representa-

tive of the class of ethers regarding which full explanation will be given when considering the official ethyl ether (p. 657).

Methyl ether is made by treating wood alcohol with sulphuric acid, and is quite popular in certain sections of Ireland as a tippie.

Methyl Salicylate.—This is the first representative of the official ethers, and is official in three forms: (a) The synthetic methyl salicylate; (b) oil of wintergreen; and (c) oil of sweet birch, the two last mentioned being natural products, consisting chiefly of the chemical above mentioned. It is more convenient, however, to consider this substance under the head of salicylic acid, hence it is deferred until that time (p. 784).

Formaldehyde (HCHO).—This, like all other aldehydes, is produced by the oxidation of the corresponding alcohol, which in this case is methyl alcohol; the reaction can be more clearly expressed as follows:



The usual method of manufacture of aldehydes from alcohol is by the oxidation with potassium dichromate and sulphuric acid, but in the case of formaldehyde oxidation by chemical means invariably leads to formic acid, instead of stopping at the half-way point of the aldehyde; hence the oxidation of wood alcohol into formaldehyde must be accomplished without the aid of chemicals, and is usually performed by the incomplete combustion of methyl alcohol. If gaseous formaldehyde is desired, this combustion is accomplished by burning the formaldehyde from a wick and covering the flame with a cone of platinum wire. Such is the principle of most of the formaldehyde lamps now largely used for purposes of disinfection. The same results can be obtained by heating wood alcohol in a flask, and as the vapor begins to rise, immersing therein a red-hot platinum spiral. The commercial method of manufacture of the chemical is by passing the vapors of wood alcohol through heated tubes containing platinum or copper spirals.

Formaldehyde is a gas of very peculiar pungent odor, and is placed on the market in aqueous solution, the commercial *formalin* being a 37 per cent. (saturated) solution of same. This is now official as *liquor formaldehydi*.

Formaldehyde is a valuable antiseptic, hence the use of formaldehyde in lamps for the disinfecting of rooms and in solution as a food preservative. The latter use of formaldehyde is prohibited, inasmuch as experiments have shown that formaldehyde, even in minute traces, injures digestion.

Formaldehyde forms with gelatinous substances insoluble compounds, and for this purpose is employed in solidifying glycerin jelly for microscopic mounts, and since hide is largely gelatin, formaldehyde can be employed for converting same into leather.

From formaldehyde many interesting syntheses have been made, the most striking of which was the condensing of same under treatment of calcium hydroxide into formose or acrose, a variety of sugar having the formula of $\text{C}_6\text{H}_{12}\text{O}_6$. This was accomplished by Lowe (1886), and gives an inkling of that wonderful method whereby plants, by the absorption of carbon dioxide from the air, grow from a seed to the large and lusty organism, the tree.

The entire details of the process are not yet fully understood, but it is generally accepted to-day that the conversion of carbon dioxide into other carbon compounds is accomplished chiefly by the intermediate product, formaldehyde.

Plants absorb carbon dioxide and water, and in these two bodies, CO_2 and H_2O , we have the elements that will form HCHO . As yet this synthesis, this building up, has not been artificially accomplished, nature holding the secret to herself. The next step in the process has been accomplished in Loew's synthesis. Formaldehyde is CH_2O . If CH_2O is multiplied by six, the result is $\text{C}_6\text{H}_{12}\text{O}_6$. Theoretically, it seems simple to convert glucose, $\text{C}_6\text{H}_{12}\text{O}_6$, into sugar, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, as explained on p. 694, and likewise to convert sugar into starch, $\text{C}_6\text{H}_{10}\text{O}_5$, and into cellulose, which has the same proportional formula and which is the main constituent of woody tissue.

The conversions have not been practically accomplished, but it can be comprehended as plausible, since the reverse operation has been done. Cotton or starch treated with sulphuric acid will yield glucose.

To sum up the process by which plants grow:

H_2O and CO_2 should give HCHO formaldehyde.

6HCHO does give $\text{C}_6\text{H}_{12}\text{O}_6$, acrose, a form of glucose.

$\text{C}_{12}\text{H}_{24}\text{O}_{12}$ ($2\text{C}_6\text{H}_{12}\text{O}_6 - \text{H}_2\text{O}$) should give $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ sugar.

$\text{C}_{12}\text{H}_{22}\text{O}_{11} - \text{H}_2\text{O}$ should give $\text{C}_{12}\text{H}_{20}\text{O}_{10}$ or $2\text{C}_6\text{H}_{10}\text{O}_5$.

($\text{C}_6\text{H}_{10}\text{O}_5$) is the formula of starch and cellulose.

Strassburger, after extensive microscopic and chemical work, decides that the method of starch formation in plants is not as simple as here outlined. He claims that very intricate intermediate products result, which is very likely, as nature rarely performs the synthesis in a direct manner. The above explanations are, therefore, merely approximate, their aim being to impress the wonderful manner in which plants live and increase on nutriment drawn almost exclusively from the air.

LIQUOR FORMALDEHYDI. Solution of Formaldehyde.

An aqueous solution containing not less than 37 per cent., by weight, of absolute Formaldehyde [$\text{H.COH} = 29.79$], an oxidation product of methyl alcohol. It should be kept in well-stoppered bottles, in a cool place, protected from light.

A clear, colorless liquid, having a pungent odor and caustic taste; its vapor acts as an irritant upon the mucous membrane.

Specific gravity: 1.075 to 1.078 at 25°C . (77°F .).

Miscible in all proportions with water and alcohol. On standing, Solution of Formaldehyde sometimes loses its transparency, the cloudiness being due to the separation of paraformaldehyde.

If Solution of Formaldehyde be evaporated over sulphuric acid, or in a vacuum, white, solid, paraformaldehyde is rapidly formed, which is insoluble in water, and which, when heated to about 100°C . (212°F .), sublimes, and between 153° and 172°C . (307.4° and 341.6°F .), melts, gaseous formaldehyde being evolved.

Solution of Formaldehyde should be neutral, or only faintly acid, to litmus-paper.

If 5 Cc. of Solution of Formaldehyde be diluted with 25 Cc. of distilled water, and 3 Cc. of silver ammonium nitrate T.S. be added, a gray precipitate of finely divided metallic silver, which often adheres in part to the sides of the test-tube as a metallic mirror, will be produced.

If to 5 Cc. of sulphuric acid in which a little salicylic acid has been dissolved 2 drops of Solution of Formaldehyde be added and the liquid very gently warmed, a permanent deep red color should immediately appear.

If 20 Cc. of Solution of Formaldehyde be evaporated to dryness on a water-bath, a white amorphous mass should remain, which, upon ignition, should not leave a residue (absence of *fixed impurities*).

If 20 Cc. of Solution of Formaldehyde, to which 2 drops of phenolphthalein T.S. have been added, be titrated with normal potassium hydroxide V.S., not more than 0.5 Cc. of the latter should be required for neutralization (absence of more than 0.1 per cent. of *formic and other acids*).

Tests for Identity.—Given above.

Impurities.—Fixed impurities, formic and other acids. See above. Chlorides, sulphates, iron, lead, copper, and calcium. Details in Part V.

Assay.—Details in Part V.

Remarks.—In view of the full statements as to formaldehyde given on the preceding page, further comments are not necessary.

Methylal (HCHOCH_3).—This substance is made by oxidizing wood alcohol with manganese dioxide and sulphuric acid and distilling the mixture. It is used as a hypnotic in doses of one to two grammes.

Formic acid, HCOOH , was discovered by Fischer and Ray in 1670, they finding same in the distillate from red ants (*Formica rufa*), hence the name. It is of interest to know that the irritating substance in the hairs of the stinging nettle, *Urtica dioica*, is chiefly formic acid.

Potassium cyanide on being cooked yields potassium formate. This, however, is not the commercial method of manufacture, and is merely suggested as a warning that pharmacists should never use heat in preparing solutions of potassium cyanide. While formic acid can be made by oxidation of methyl alcohol, it is usually prepared by heating oxalic acid with glycerin, the distillate being practically pure formic acid.

Formic acid has very little medicinal and pharmaceutic use, but it is interesting to note that it is a reducing agent, being capable of producing the separation of metallic silver from the solution of silver nitrate. It is the only acid of the series possessing reducing power.

The Methylamines.—Of these, the only one of pharmaceutic value is *trimethylamine*, $(\text{CH}_3)_3\text{N}$. This body was introduced into the market under the name of *propylamine*, it being an isomere of that body which has the formula $\text{C}_3\text{H}_7\text{NH}_2$. It is found in small traces in arnica root, and a considerable quantity of the brine used for pickling herring. Its commercial source is beet sugar residues, and it had considerable vogue for rheumatism, but its disagreeable taste prevented its general use. Dose, 2 to 4 drops.

CHAPTER XXXVII

ETHANE DERIVATIVES

THE derivatives of ethane of pharmaceutic value are:

Ethyl chloride, U. S. P.....	$\text{C}_2\text{H}_5\text{Cl}$.
Alcohol, U. S. P.....	$\text{C}_2\text{H}_5\text{OH}$.
Ether, U. S. P.....	$(\text{C}_2\text{H}_5)_2\text{O}$.
Ethyl sulphate.....	$(\text{C}_2\text{H}_5)_2\text{SO}_4$.
(in ethereal oil, U. S. P.).	
Ethyl nitrite.....	$\text{C}_2\text{H}_5\text{NO}_2$.
(in spirits of nitrous ether, U. S. P.).	
Ethyl acetate, U. S. P.....	$\text{C}_2\text{H}_5\text{C}_2\text{H}_3\text{O}_2$.
Ethyl carbamate, U. S. P.....	$\text{C}_2\text{H}_5\text{NH}_2\text{CO}_2$.
(described among the urea derivatives).	
Aldehyde.....	CH_3CHO .
Paraldehyde, U. S. P.....	$(\text{CH}_3\text{CHO})_3$.
Chloral hydrate, U. S. P.....	$\text{CCl}_3\text{CHO} + \text{H}_2\text{O}$.
Chloral formamide, U. S. P.....	$\text{CCl}_3\text{CH}(\text{OH})(\text{NHCOH})$.
Acetic acid, U. S. P.....	CH_3COOH .
Oxalic acid.....	$(\text{COOH})_2$.

ÆTHYLIS CHLORIDUM. Ethyl Chloride.

A haloid derivative [Monochlor-Ethane, $\text{C}_2\text{H}_5\text{Cl}$ =64.00], prepared by the action of hydrochloric acid gas upon absolute ethyl alcohol.

On account of its extreme volatility, it should be preserved in hermetically sealed glass tubes, and kept in a cool place, remote from lights or fire.

A colorless, mobile, very volatile liquid, having a characteristic, rather agreeable odor, and a burning taste.

Specific gravity: 0.918 at 8° C. (46.4° F.).

Ethyl chloride is slightly soluble in water, readily soluble in alcohol.

It boils at a temperature of 12.5° to 13° C. (54.5° to 55.4° F.), and at its ignition temperature burns with a smoky, green-edged flame, with the production of gaseous hydrochloric acid.

When liberated, at ordinary room-temperatures, from its sealed glass tube, Ethyl Chloride vaporizes at once; the gas is very inflammable, and consequently it should not be used in proximity to a gas-flame or fire.

If 10 Cc. of Ethyl Chloride, while cold, be dissolved in alcohol and a few drops of silver nitrate T.S. be added, no turbidity should be produced (absence of *hydrochloric acid*).

If 10 Cc. of Ethyl Chloride be agitated with 10 Cc. of cold water, and the supernatant stratum of Ethyl Chloride be evaporated spontaneously, and if a few drops of potassium dichromate T.S. be added to the remaining aqueous liquid, followed by some diluted sulphuric acid, and the mixture be boiled, no odor of aldehyde should be developed, and a greenish or purplish color should not be produced in the liquid (absence of *alcohol*).

On allowing Ethyl Chloride to evaporate from clean, odorless blotting paper which has been saturated with it, no unpleasant odor should remain upon the paper (absence of *sulphur compounds*, etc.).

Remarks.—As above defined, ethyl chloride is made by treating alcohol with hydrochloric acid gas. It can also be made, as already explained, as all other halogen derivatives of the hydrocarbons, by treatment of the appropriate hydrocarbon (in this case, ethane) with the proper halogen (in this case, chlorine).

Ethyl chloride is so volatile that it can be kept in its liquid form only when under heavy pressure in sealed tubes. It is used, under such trade names as *kelene*, as a local anesthetic (as described under Rhigolene, p. 637), and is now being employed either by itself or in combination with chloroform or ether as a general anesthetic.

Ethyl hydroxide (C_2H_5OH) has been known from the earliest times in the form of alcoholic liquids, one of the first references being the well-known story of Noah and his vineyard.

The isolation was first performed by Rhazes (tenth century), and the name alcohol was first applied in the thirteenth century, the name being of Arabic origin.

Passing over the several syntheses of alcohol (such as treating ethyl chloride with moist silver oxide), it may be said that the sole commercial origin of alcohol is by the fermentation of substances containing starch or sugar. If dilute watery preparations of starch and sugar are allowed to stand, the spores of the yeast plant, *Saccharomyces cerevisiae*, present in the air, locate in same and begin to grow, the process of the plant's assimilation producing in saccharine liquid the chemical change called *fermentation*. It was formerly considered that the chemical change could be induced only by the actual process of plant life, but recent investigations have shown that the fermentation is entirely due to the chemical action of the protoplasm of yeast, and at present, in the brewing of beer and other malt liquids, it is preferred to use the expressed liquid from the destroyed yeast plant rather than the living plant itself. The reaction of the fermentation expressed in simplest form is as follows:



but the real process is far more complicated, as is evidenced by the large number of side-products of fermentation, such as glycerin, succinic acid, and amylic alcohol, invariably found in the fermented liquid.

In order to obtain alcohol it is necessary to subject the fermented liquid (usually called the *mash*) to distillation.

Of the many alcoholic liquors employed as beverages, some are distilled products and others are the clarified, undistilled products of fermentation. Among the important alcoholic liquids are wines, beer, whisky, and brandy.

Wines are made by the fermentation of the juice of grapes and eventual clarification of the same. Wines are grouped by the pharmacœia into *white wine*, U. S. P. (*vinum album*), which is made by fermenting the juice of the grape, freed from the skins; *red wine*, U. S. P. (*vinum rubrum*), obtained by the fermentation of the juice of the red grapes in the presence of the skins.

VINUM ALBUM. White Wine.

An alcoholic liquid, made by fermenting the juice of fresh grapes, the fruit of *Vitis vinifera* Linné (Fam. *Vitaceæ*), freed from seeds, stems, and skins, and subjected to the usual cellar-treatment for fining and aging.

When White Wine is prescribed without further specification, it is recommended that a dry White Wine of domestic production be employed.

White Wine should be preserved in well-closed casks filled as full as possible, or in well-stoppered bottles, in a cool place.

A pale amber-colored or straw-colored liquid, having a pleasant odor free from yeastiness, and a fruity, agreeable, slightly spirituous taste without excessive sweetness or acidity.

The specific gravity, at 15.6° C. (60° F.), should not be less than 0.990 nor more than 1.010.

If a portion of White Wine be evaporated, the residue, when dried during twelve hours on a water-bath, should amount to not less than 1.5 nor more than 3 per cent.; this residue, ignited at a low temperature and burned gradually to whiteness, moistened with a small portion of ammonium carbonate T.S., and again carefully ignited, should weigh not less than 0.14 Gm. nor more than 0.26 Gm. for each 100 Cc. of White Wine tested.

To neutralize 50 Cc. of White Wine should require not less than 3 nor more than 5.2 Cc. of normal potassium hydroxide V.S. (limit of *free acid*), litmus T.S. being used as indicator.

If 10 Cc. of White Wine be diluted with an equal volume of water, and treated with 5 drops of ferric chloride T.S., only a faint, greenish-brown color should make its appearance (absence of more than traces of *tannic acid*).

If 75 Cc. of White Wine be acidified with 5 Cc. of diluted sulphuric acid (1 in 3) and thoroughly shaken in a separator with a mixture of equal parts of petroleum benzin and ether, and if the solvent, after separation, be transferred to a porcelain dish, allowed to evaporate spontaneously, and the residue dissolved in 3 Cc. of water, the solution should not have a sweet taste (absence of *saccharin*), nor should it give a violet color upon the addition of a diluted solution of ferric chloride (1 in 200) (absence of *salicylic acid*).

Tested by the following method, White Wine should be found to contain not less than 7 per cent. nor more than 12 per cent., by weight (equivalent to 8.5 per cent. to 15 per cent. by volume), of absolute alcohol.

Take the specific gravity (to four decimals) of a sufficient portion of the White Wine carefully measured at the temperature of 15.6° C. (60° F.), evaporate the Wine in a tared dish to one-third of its original weight, cool, and add water until the liquid measures its original volume at 15.6° C. (60° F.); then take the specific gravity (to four decimals) again. The difference between the two specific gravities deducted from 1.0000 corresponds to the specific gravity of an alcohol containing the same percentage of absolute alcohol, by weight, or volume as the Wine under examination, the corresponding percentage being ascertained by referring to the alcohol tables.

Tests for Identity.—Given above.

Impurities.—Free acid, tannic acid, salicylic acid, saccharin. Details above.

Estimation of Strength.—Specific gravity process given above.

VINUM RUBRUM. Red Wine.

An alcoholic liquid, made by fermenting the juice of fresh red-colored grapes, the fruit of *Vitis vinifera* Linné (Fam. *Vitaceæ*), in presence of their skins, and subjected to the usual cellar-treatment for fining and aging.

When Red Wine is prescribed without further specification, it is recommended that a dry Red Wine of domestic production be employed.

Red Wine should be preserved in well-closed casks filled as full as possible, or in well-stoppered bottles, in a cool place.

A deep red liquid, having a pleasant odor free from yeastiness, and a fruity, moderately astringent, pleasant, and slightly acidulous taste, without excessive sweetness or acidity.

The specific gravity, at 15.6° C. (60° F.), should be not less than 0.989 nor more than 1.010.

If a portion of Red Wine be evaporated, the residue, when dried during twelve hours on a water-bath, should amount to not less than 1.6 per cent. nor more than 3.5 per cent.; this residue, ignited at a low temperature and burned gradually to whiteness, moistened with a small portion of ammonium carbonate T.S., and again carefully ignited, should weigh not less than 0.22 Gm. nor more than 0.34 Gm. for each 100 Cc. of Red Wine tested.

With lead acetate T.S. Red Wine forms a heavy precipitate which may vary in color from bluish-green to green.

If 50 Cc. of Red Wine be treated with a slight excess of ammonia water, the liquid should acquire a green or brownish-green color; if it be then well shaken with 25 Cc. of ether, the greater portion of the ethereal layer removed, and evaporated in a porcelain dish with an excess of acetic acid and a few fibers of uncolored silk, the latter should not acquire a crimson or violet color (absence of *red aniline colors*).

If 25 Cc. of Red Wine, heated to about 45° C. (113° F.), be well agitated with 25 Gm. of manganese dioxide, the liquid filtered off and acidulated with hydrochloric acid, it should not acquire a red color (absence of *acid fuchsine*).

Tests for Identity.—Given above.

Impurities.—Free acid, tannic acid, salicylic acid, and saccharin, as given under White Wine. Red aniline colors and acid fuchsin. Details above.

Estimation of Strength.—As given under White Wine.

These wines are directed by the pharmacopœia to contain not less than 7 nor more than 12 per cent. of alcohol by weight, this being determined by taking the specific gravity process given above. The process is an ingenious one, and gives fairly accurate results if careful note is made of the temperature.

The process can be explained by actual figures:

The specific gravity of a sample of the wine before evaporation was 0.9943

The specific gravity of a sample of the wine after evaporation was 1.0109

The difference was 0.0166

1.000—0.0166=0.9834 { The specific gravity of a sample of alcohol of the same strength as the wine.

Reference to the alcohol table of the pharmacopœia (U. S. P. VIII., p. 603) shows—

that alcohol 10 per cent. by weight has specific gravity 0.9839.

that alcohol 11 per cent. by weight has specific gravity 0.9826.

Hence, the wine contained between 10 per cent. and 11 per cent. alcohol (by weight).

The pharmacopœia very wisely takes the stand in favor of the domestic wines, those of good quality being much better than imported wines at the same price.

White wine is used as a menstruum in making official wines, and both wines are administered as stimulants.

Champagne, though unofficial, deserves passing comment. This represents white wine containing from 8 to 9 per cent. of alcohol, which is carbonated with carbon dioxide generated during the process of fermentation. The sweetened wine with a minute quantity of yeast is placed in tightly stoppered bottles, which are inverted on a rack and left in a room, the temperature of which favors fermentation. When the fermentation has reached the proper stage—which is determined by experience—the wine is left to cool for several months, during which time the sediment collects in the neck of the bottle while the gas collects at the inverted bottom. When completely settled, an expert workman passes from bottle to bottle, drawing out the corks for a period long enough to remove the sediment without serious loss of the other contents. The product is then corked securely and sent to market. It can readily be seen that the operation of removing the sediment is exceedingly delicate, and can be accomplished only by failure to produce this result on a large number of bottles. This loss plus the loss of breakage of bottles during process of fermentation gives the real reason for the high cost of champagne.

Beer.—This well-known unofficial is the product of the fermentation of malt, hops being added to give the product a characteristic taste. The ordinary lager beer contains 3 to 4 per cent. of alcohol; the dark, heavy Munich beer contains 8 per cent. of alcohol, while the so-called Berlin or Weiss beer contains only 2 per cent., which amount is too small to effect complete preservation of the product, hence it must be fresh when dispensed.

SPIRITUS FRUMENTI. Whisky.

An alcoholic liquid obtained by the distillation of the mash of fermented grain—such as Indian corn, rye, wheat, and barley, or their mixtures.

An amber-colored liquid, having a distinctive odor and taste, and a slightly acid reaction.

Whisky should be at least four years old.

Its specific gravity should not be more than 0.945 nor less than 0.924 at 15.6° C. (60° F.), corresponding, approximately, to an alcoholic strength of 37 to 47.5 per cent., by weight, or 44 to 55 per cent., by volume, of absolute alcohol.

If 100 Cc. of Whisky be very slowly evaporated in a tared dish on a water-bath, the last portions volatilized should not have a harsh or disagreeable odor (absence of more than a trace of *fusel oil* from grain); and the residue, when dried at 100° C. (212° F.), should not weigh more than 0.5 Gm. This residue should have no sweet or distinctly spicy taste (absence of *added sugar*, *glycerin*, and *aromatic substances*), and it should almost completely dissolve in 10 Cc. of cold water, forming a solution which is colored not deeper than light green by a few drops of ferric chloride T.S. diluted with 10 volumes of water (absence of more than traces of *oak tannin* from casks).

If 50 Cc. of Whisky be shaken vigorously in a stoppered flask with 25 Gm. of kaolin, and, after standing half an hour, be filtered, the color of the filtrate should not be much lighter than that of the Whisky before treatment.

To render 100 Cc. of Whisky distinctly alkaline to litmus-paper, not more than 1.2 Cc. of normal potassium hydroxide V.S. should be required (limit of *free acid*).

Tests for Identity.—Given above.

Impurities.—Excess of fusel oil, added sugar, glycerin, aromatic substances, excess of tannin, excess of free acid. Details given above.

Added caramel coloring (the filtration through kaolin test as given above).

Remarks.—This is a product of distillation of the mash of fermented grain, usually of a mixture of corn, wheat, and rye, and at least four years old. Whisky contains from 37 to 47.5 per cent. of alcohol by weight. It should be four years old, because some of the side-products (fusel oil, for example) are deleterious, but on aging four years this fusel oil will have combined with the other constituents of whisky to produce an aromatic substance which in itself is practically harmless. (21)

SPIRITUS VINI GALLICI. Brandy.

An alcoholic liquid obtained by the distillation of the fermented, unmodified juice of fresh grapes.

A pale amber-colored liquid, having a distinctive odor and taste, and a slightly acid reaction.

Brandy should be at least four years old.

Its specific gravity should be not more than 0.941 nor less than 0.925 at 15.6° C. (60° F.), corresponding, approximately, to an alcoholic strength of 39 to 47 per cent., by weight, or 46 to 55 per cent., by volume, of absolute alcohol.

Tests for Identity.—Given above.

Impurities.—Excess of fusel oil, added sugar, glycerin, aromatic substances, oak tannin, caramel coloring, exactly as given under Whisky. Excess of free acid, estimation as in whisky except 100 Cc. made alkaline with 1 Cc. normal potassa solution.

Remarks.—Brandy is obtained by the distillation of the fermented, unmodified juice of fresh grapes, and at least four years old. It should contain 39 to 47 per cent. by weight, of alcohol, and is directed by the pharmacopœia to be kept

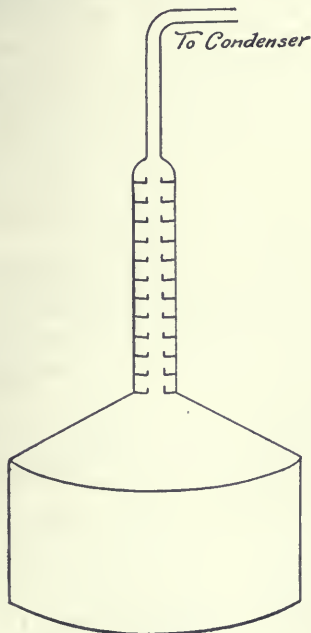


Fig. 219.—Column distilling apparatus.



Fig. 220.—Glass dephlegmator.

four years in order to age it, as explained above. Both whisky and brandy are used medicinally as stimulants, being given in doses of about half an ounce.

Alcohol is obtained by the redistillation of whisky, the fusel oil, glycerin, and other extraneous matter being separated from same by the process called *dephlegmation*. This long word had been bequeathed us by the alchemists, whose favorite treatment of a substance was to submit it to distillation. If a portion thereof was volatile, the same was called *spirit*, while the less volatile portion which was left behind was called *phlegm*. To produce this spirit (alcohol) it was necessary to remove same from the phlegm and the process was called *dephlegmation*. This is accomplished by heating the whisky in an appropriate still body, and making the vapors rise through a tall tower or column before diverting same into the condensing worm. This tower or column apparatus is generally provided with a series of projections, which condense the less volatile portions, permitting the passage only of the pure alcohol. A glass dephlegmator for smaller operations of the laboratory is shown in Fig. 220. Alcohol is official in three different forms:

ALCOHOL. Alcohol.

A liquid composed of about 92.3 per cent., by weight, or about 94.9 per cent., by volume, of absolute Ethyl Alcohol [$C_2H_5.OH = 45.70$], and about 7.7 per cent., by weight, of water. It should be kept in well-closed vessels, in a cool place, remote from lights or fire.

A transparent, colorless, mobile and volatile liquid, of a slight, agreeable odor, and a burning taste.

Specific gravity: about 0.816 at 15.6° C. (60° F.), the standard temperature for Alcohol, or 0.809 at 25° C. (77° F.).

Miscible with water in all proportions, and without any trace of cloudiness; also miscible with ether or chloroform.

It is readily volatilized, even at low temperatures, and boils at 78° C. (172.4° F.). It is inflammable and burns with a pale blue, smokeless flame.

It should not affect the color of blue or red litmus-paper previously moistened with water.

If 50 Cc. of Alcohol be evaporated in a clean glass vessel, no color or weighable residue should remain.

If 10 Cc. of Alcohol be mixed with 5 Cc. of water and 1 Cc. of glycerin, and the mixture allowed to evaporate spontaneously from a piece of clean, odorless blotting paper, no foreign odor should become perceptible when the last traces of the Alcohol leave the paper (absence of fusel oil constituents).

If 25 Cc. be allowed to evaporate spontaneously in a porcelain evaporating dish, carefully protected from dust, until the surface of the dish is barely moist, no red or brown color should be produced upon the addition of a few drops of colorless, concentrated sulphuric acid (absence of amyl alcohol, or non-volatile, carbonizable, organic impurities, etc.).

If 10 Cc. of Alcohol be mixed in a test-tube with 5 Cc. of potassium hydroxide T.S., the liquid should not at once assume a yellow color (absence of aldehyde or oak tannin).

If 20 Cc. of Alcohol be shaken in a clean, glass-stoppered vial with 1 Cc. of silver nitrate T.S., the mixture should not become more than faintly opalescent, or acquire more than a faint brownish tint when exposed for six hours to diffused daylight (limit of organic impurities, amyl alcohol, aldehyde, etc.).

Methyl Alcohol Test.—Into a test-tube of the capacity of about 40 cubic centimeters, 1 Cc. of Alcohol or spirit to be tested should be poured, and, if it be undiluted, enough distilled water added to make the liquid measure 10 Cc. If the alcohol be already diluted, a correspondingly larger volume of it should be taken and diluted to 10 Cc., so that the proportion of Alcohol in the liquid shall not be more than about 10 per cent., by volume. A copper wire spiral (made by winding 1 meter of No. 18 clean copper wire closely around a glass rod 7 millimeters thick, making a coil about 3 centimeters long, the end of the wire being formed into a handle) should be heated to redness in a flame free from soot, and plunged steadily quite to the bottom of the liquid in the test-tube and held there for a second or two, then withdrawn and dipped into water to cool. This treatment with red-hot copper should be

repeated five or six times, immersing the test-tube in cold water to keep down the temperature of the liquid. The contents of the test-tube should now be filtered into a wide test-tube and boiled very gently. If the odor of acetaldehyde be perceptible, the boiling is to be continued until the odor ceases to be distinguished clearly. The liquid is now cooled, and to it should be added 1 drop of a solution containing 1 part of resorcinol in 200 parts of water. A portion of this liquid is then poured cautiously into a second tube containing pure sulphuric acid, in such a way that the two liquids shall not mix, the tube being held in an inclined position; this tube is allowed to stand for three minutes, and then slowly rotated. No rose-red ring should show at the line of contact of the two layers (absence of more than 2 per cent. of *methyl alcohol*).

Tests for Identity.—Given above.

Impurities.—Fusel oil constituents; amyl alcohol; non-volatile, carbonizable, organic impurities; aldehyde; oak tannin; nor more than 2 per cent. methyl alcohol. Details above.

Remarks.—This represents the most concentrated form of ethyl hydrate which can be produced by the simple process of dephlegmation. It contains 92.3 per cent., by weight (94.9 per cent., by volume), of ethyl hydrate, and is practically the *deodorized alcohol* of the pharmacopœia of 1890.

It is very important to be able readily to determine the strength of any individual sample of alcohol on hand, and this is usually accomplished by means of that form of hydrometer called the alcoholometer (already described on p. 50), which is usually provided with a scale which not merely reads its specific gravity, but also the actual percentage of alcohol in the sample. Of course, the strength can be ascertained by taking the specific gravity in any other manner, in which event, however, reference must be had to the alcohol table found in the back of the pharmacopœia.

It will be noticed that the present pharmacopœia includes a test for wood alcohol which consists of oxidizing the methyl alcohol to formaldehyde by plunging therein a red-hot copper spiral (see p. 646), and detecting the formaldehyde thus produced by the resorcinol-sulphuric acid test.

ALCOHOL ABSOLUTUM. Absolute Alcohol.

Ethyl Alcohol [$C_2H_5.OH=45.70$], containing not more than 1 per cent., by weight, of water. It should be kept in well-stoppered bottles or tin cans, in a cool place, remote from lights or fire.

A transparent, colorless, mobile, and volatile liquid, of a characteristic, rather agreeable odor, and a burning taste. Very hygroscopic.

Specific gravity: not higher than 0.797 at 15.6° C. (60° F.) nor 0.790 at 25° C. (77° F.).

In other respects Absolute Alcohol has the properties, and should respond to the reactions and tests, of Alcohol (see *Alcohol*).

Remarks.—This is a liquid containing not less than 99 per cent. of ethyl hydroxide, and is made from ordinary alcohol by redistillation from lime. In order to get same "absolutely absolute," which is sometimes necessary in very careful chemical work, it is mixed with metallic sodium, which absorbs all the water (forming sodium hydroxide), and also some of the alcohol (forming sodium alcoholate, $C_2H_5.ONa$), and then distilling into a flask closely fitted to the condenser, and with orifices for air closed by means of calcium chloride drying tubes.

To tell whether absolute alcohol is completely dehydrated is

usually accomplished by throwing into same a piece of exsiccated copper sulphate. This is of grayish-white color, and if there is any water in the alcohol, the copper salt absorbs same, becoming partly crystallized, and assumes a blue tint.

ALCOHOL DILUTUM. Diluted Alcohol.

A liquid composed of about 41.5 per cent., by weight, or about 48.9 per cent., by volume, of absolute Ethyl Alcohol [$C_2H_5.OH=45.7$], and about 58.5 per cent., by weight, of water. It should be kept in well-closed vessels, in a cool place, remote from lights or fire.

Recipe.—**Alcohol**, five hundred cubic centimeters..... 500 Cc.
Distilled Water, five hundred cubic centimeters..... 500 Cc.

Mix them.

If the two liquids be measured at the temperature of 25° C. (77° F.), the mixture, when cooled to the same temperature, will measure about 970 Cc.

Diluted Alcohol may also be prepared in the following manner:

Alcohol, four hundred and eight grammes..... 408 Gm.
Distilled Water, five hundred grammes..... 500 Gm.

Mix them.

Diluted Alcohol has a specific gravity of about 0.936 at 15.6° C. (60° F.), the standard temperature for Alcohol, and about 0.930 at 25° C. (77° F.).

It should respond to the reactions and tests given under *Alcohol*.

RULES FOR MAKING AN ALCOHOL OF ANY REQUIRED LOWER PERCENTAGE FROM AN ALCOHOL OF ANY GIVEN HIGHER PERCENTAGE.

I. By Volume.—Designate the volume-percentage of the stronger alcohol by V , and that of the weaker alcohol by v .

Rule.—Mix v volumes of the stronger alcohol with distilled water to make V volumes of product. Allow the mixture to stand until full contraction has taken place and until it has cooled, then make up the deficiency in the V volumes by adding more distilled water.

Example.—An alcohol of 30 per cent., by volume, is to be made from an alcohol of 94.9 per cent., by volume: Take 30 volumes of the 94.9 per cent. alcohol and add enough distilled water to produce 94.9 volumes.

II. By Weight.—Designate the weight-percentage of the stronger alcohol by W , and that of the weaker alcohol by w .

Rule.—Mix w parts by weight of the stronger alcohol with distilled water to make W parts by weight of product.

Example.—An alcohol of 50 per cent., by weight, is to be made from an alcohol of 92.3 per cent., by weight: Take 50 parts, by weight, of the 92.3 per cent. alcohol and add enough distilled water to produce 92.3 parts, by weight.

Remarks.—Diluted alcohol, U. S. P., is made by mixing equal volumes of alcohol and water, and since U. S. P. alcohol is 91 per cent., by weight, of ethyl hydroxide, the dilute alcohol is 41 per cent., by weight, of the same chemical. This is shown in the following figures:

100 Cc. of 91 per cent. alcohol (sp. gr. 0.820) weighs 82 Gm.
 plus 100 Cc. or 82 Gm. 91 per cent. alcohol
 100 Cc. or 100 Gm. water

makes 200 Cc. or 182 Gm. of diluted alcohol.

The 82 Gm. 91 per cent. alcohol contain 74.62 Gm. absolute alcohol.

$$(82 \times 0.91 = 74.62).$$

Hence the 182 Gm. diluted alcohol contain the same—

74.62 Gm. absolute alcohol.

What percentage of 182 is 74.62?

$$\frac{74.62}{182.00} \times 100 \text{ per cent., or } 41 \text{ per cent.}$$

Note that 100 Cc. alcohol and 100 Cc. water do not make 200 Cc. diluted alcohol, and this variation from the theoretic is due to the interesting fact that when alcohol and water are mixed, contraction occurs, as explained on p. 122.

It is important to know that diluted alcohol, U. S. P., is not the "proof spirit" of the Internal Revenue Department of the government; the latter means 50 per cent. by volume of ethyl hydrate, while the dilute alcohol, U. S. P., is 48.6 per cent. by volume. As is usual with commercial standards, the Internal Revenue grading is wholly unscientific.

In choosing 50 per cent. by volume as "proof," possibly a correct standard was made, as it is assumed that most whisky is about that strength. However, the strength, either above or below this point, is designated in a most unfortunate manner, viz., by saying that sample is "ten below proof" or "twenty above proof." Twenty above proof does not mean 70 per cent. by volume, but indicates that 100 parts of such 20 above proof sample are to be diluted with water enough to make 120 parts in order to make it "proof." In other words, "20 above proof" means 60 per cent. by volume.

Still more unfortunate is it that British excise "proof" is not the same as the standard of the Internal Revenue of our country, but represents 50 per cent., by weight, of absolute alcohol.

The rule for alcohol mixing given by the pharmacopœia and inserted above is simply the expression, in another form, of the rule of alligation given on p. 65.

Ethyl Oxide ($C_2H_5)_2O$.—This product is official as *Æther*.

ÆTHER. Ether.

A liquid composed of about 96 per cent., by weight, of absolute Ether or Ethyl Oxide [$(C_2H_5)_2O = 73.52$] and about 4 per cent. of alcohol containing a little water. Ether should be kept in partially filled, well-stoppered containers, preferably tin cans, in a cool place, remote from lights or fire.

A transparent, colorless, mobile liquid, having a characteristic odor and a burning and sweetish taste.

Specific gravity: 0.716 to 0.717 at 25° C. (77° F.).

Soluble in about 10 times its volume of water at 25° C. (77° F.), with slight contraction of volume. Miscible, in all proportions, with alcohol, chloroform, petroleum benzin, benzene, fixed and volatile oils.

Ether boils at about 35.5° C. (96° F.), and it should, therefore, boil when a test-tube, containing some broken glass and half filled with it, is held for some time closely grasped in the hand.

Ether is highly volatile and inflammable. Its vapor, when mixed with air and ignited, explodes violently.

The color of light blue litmus-paper moistened with water should not be changed to red when the paper is immersed in Ether for ten minutes.

Upon evaporation, Ether should leave no *residue*.

If 10 Cc. of it be poured, in portions, upon clear, odorless blotting-paper, and allowed to evaporate spontaneously, no *foreign odor* should become perceptible when the last traces of Ether leave the paper.

When 20 Cc. of Ether are shaken, in a graduated tube, with 20 Cc. of water, just previously saturated with Ether, the ether-layer, upon separation, should measure not less than 19.2 Cc. (absence of an *undue amount of alcohol or water*).

If 10 Cc. of Ether be shaken occasionally, during one hour, with 1 Cc. of potassium hydroxide T.S., no color should be developed in either liquid (absence of *aldehyde*).

Tests for Identity.—Given above.

Impurities.—Excess of alcohol or water; aldehyde. Details above.

Remarks.—While ether was known to Raymond Lully (1253–1315), the present method of manufacture was devised by Valerius Cordus in the middle of the fifteenth century. It is made by treating alcohol with sulphuric acid and distilling immediately.

As explained on p. 631, the reaction may be simply expressed as being produced by taking water away from alcohol, sulphuric acid acting as the dehydrating agent. This theory of ether formation proposed by Fourcroy and Vauquelin (1797) was accepted until later investigations proved that while true as to ultimate results, the actual process was not so simple as the mere removal of water by mechanical means. The factor contributing to the rejection of the theory was the fact that if the water was absorbed by the sulphuric acid, it would be closely retained by same, when, as a matter of fact, it is noted that a portion of the water distils over with the ether.

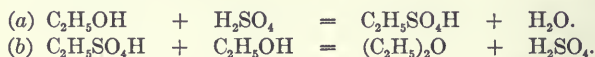
The next theory was the “contact theory” of Mitscherlich, that the ether production was due to catalysis, the latter word a term found very convenient by chemists of the first half of the last century for explaining chemical changes which were not really understood.

The discovery of the real process was made by Liebig (1839), who proved that, during the formation of ether, an intermediate product, ethylsulphuric acid, $\text{SO}_2 < \begin{smallmatrix} \text{OH} \\ \text{OC}_2\text{H}_5 \end{smallmatrix}$, is produced.

This is sometimes called “*sulphovinic acid*,” and forms salts, one of which, sodium sulphovinate, had some medical prominence some years since, being recommended by Dr. Rabuteau as a purgative in 25-Gm. doses.

Liebig’s discovery of ethyl-sulphuric acid was supplemented by the researches of Williamson (1850), whose complete theory of ether formation is now accepted as truth.

According to this, alcohol treated with sulphuric acid forms ethyl-sulphuric acid and water (equation *a*). This ethyl-sulphuric acid comes in contact with more alcohol, liberating ether and sulphuric acid (equation *b*):



It will be noted that the above reaction finishes with as much acid as it began, and this is a fact, a small quantity of sulphuric acid being capable of etherizing a large quantity of alcohol.

The only reason why the same sulphuric acid cannot be used indefinitely is the fact that the acid finally becomes so greatly diluted with water that it is incapable of further chemical action.

As to the practical details of ether formation, the sulphuric acid is placed in a suitable container (still body), gently warmed, and upon it the alcohol is allowed slowly to trickle. Contact of the alcohol with the acid is followed by the formation of ether, which is immediately distilled. The distillate usually contains about 74 per cent. of ethyl oxide, and in this form was official in the pharmacopœia of 1880. The greater portion of the alcohol can be removed by cautious distillation, and ether, U. S. P., which was called *æther fortior* in

U. S. P. 1880, is directed to contain at least 96 per cent. of ethyl oxide.

Ether is a clear, limpid liquid of specific gravity 0.716, boiling at 37° C., and as it is exceedingly volatile, must be kept in tightly closed bottles away from a flame. It is used in pharmacy as a solvent, being the menstruum formerly directed in the preparation of the oleoresins.

Its medicinal use is as an anesthetic, while in minute quantities it acts as a sedative.

Dose.—1 Cc. (15 minims).

Syrup of ether of the French Codex is well adapted for administration of ether as a sedative. This is made by agitating 500 parts of syrup with 30 parts of ether in a bottle provided with an orifice at the bottom (the Thudicum nasal douche bottle, Fig. 221, with rubber tube replaced by a cork). Ether is macerated with the syrup for five or six days with occasional agitation; then it is allowed to stand, and the clear syrup drained off from the ethereal mixture, which floats on the surface. This is the only satisfactory way of making syrup of ether.

Schleich's solution was introduced some years ago as the safest form of anesthetic.

There are three forms of the solution recommended by Dr. Schleich for anesthesia for short, moderate, or long periods respectively:

- I. Chloroform 45, ether 180, petroleum ether 15. Boiling-point, 38° C.
(B. P. 60°–65°)
- II. Chloroform 45, ether 150, petroleum ether 15. Boiling-point, 40° C.
- III. Chloroform 30, ether 80, petroleum ether 15. Boiling-point, 42° C.

It is an open question as to whether such mixture even possesses a definite boiling-point.

In dispensing this the pharmacist must be careful to use petroleum ether and not commercial benzin. For similar purposes is the "*A. C. E. Mixture*," which consists of alcohol, 1 fluidounce; chloroform, 2 fluidounces, and ether, 3 fluidounces. This is very popular in English hospitals.

Two official preparations of ether are *spirit of ether* and *compound spirit of ether*, both of which are described under Spirits (p. 177).

Four esters of ethyl hydrate are found in official preparations: *ethyl nitrite*, in spirit of nitrous ether; *ethyl acetate*, official as acetic ether; *ethyl sulphate*, in ethereal oil; and *ethyl carbamate* or urethane.

SPIRITUS ÆTHERIS NITROSI. Spirit of Nitrous Ether.

An alcoholic solution of Ethyl Nitrite [$\text{NO} \cdot \text{OC}_2\text{H}_5 = 74.51$], yielding, when freshly prepared and tested by the process given below, not less than 4 per cent. of ethyl nitrite.

<i>Recipe.</i> —Sodium Nitrite, one hundred grammes.....	100.0 Gm.
Sulphuric Acid, forty cubic centimeters.....	40.0 Cc.
Monohydrated Sodium Carbonate, six-tenths of a gramme.....	0.6 Gm.
Potassium Carbonate, completely deprived of water by drying, three grammes.....	3.0 Gm.
Alcohol,	
Water, each, a sufficient quantity.	

Mix the Sulphuric Acid with one hundred and twenty cubic centimeters of Water, cool the liquid, add eighty-five cubic centimeters of Alcohol previously diluted with an



Fig. 221.—Nasal douche bottle.

equal volume of Water, and introduce the solution into a 1000-Cc. flask, surrounded by a mixture of ice and water. Dissolve the Sodium Nitrite in *two hundred and eighty cubic centimeters* of Water, filter, and, having poured the filtrate into a separatory funnel, allow the liquid to slowly drop into the flask containing the acid mixture. When all has been added and the reaction is complete, allow any crystals which may have formed to settle at the bottom of the flask, and decant the cold mixture of ethyl nitrite and aqueous solution quickly to the previously cleaned separatory funnel, and draw off and discard the aqueous liquid. Wash the separated ethyl nitrite, first, with *twenty cubic centimeters* of ice-cold Water, and then remove any traces of acid by washing it with *fifteen cubic centimeters* of ice-cold Water, in which the Monohydrated Sodium Carbonate has previously been dissolved. Carefully separate the ethyl nitrite from the aqueous liquid, and agitate it in a well-stoppered vial with the Potassium Carbonate to remove traces of water. Then cool the liquid, decant, and pour the ethyl nitrite immediately into a tared bottle containing *five hundred grammes* of Alcohol. Ascertain the weight of the ethyl nitrite poured into the Alcohol by noting the increase in weight of the tared bottle and contents, and then add enough Alcohol to make the mixture weigh *twenty-two times* the weight of the ethyl nitrite added. Lastly, transfer the product to small, well-stoppered, dark amber-colored vials, and keep these in a cool place, remote from lights or fire.

A clear, mobile, volatile, and inflammable liquid of a pale yellowish or faintly greenish-yellow tint, having a fragrant, ethereal, and pungent odor, free from acidity, and a sharp, burning taste.

Specific gravity: about 0.823 at 25° C. (77° F.).

When freshly prepared, or even after being kept for some time with but little exposure to light and air, it is neutral to litmus-paper. When long kept, or after having been freely exposed to air and light, it acquires an acid reaction, but it should not effervesce when a crystal of potassium bicarbonate is dropped into it.

If a test-tube be half filled with the Spirit, and put into a water-bath heated to 65° C. (149° F.) until it has acquired this temperature, the Spirit should boil distinctly upon the addition of a few small pieces of broken glass.

If 10 Cc. of the Spirit be mixed with 5 Cc. of potassium hydroxide T.S., previously diluted with 5 Cc. of water, the mixture will assume a yellow color which should not turn decidedly brown within twelve hours (limit of *aldehyde*).

ASSAY FOR ETHYL NITRITE

Transfer about 30 Gm. of the Spirit of Nitrous Ether, which has been previously shaken with 0.5 Gm. of potassium bicarbonate, to a tared 100-Cc. measuring flask, and weigh it accurately. Add sufficient alcohol to bring the volume to exactly 100 Cc., and mix thoroughly. Introduce into a nitrometer (see p. 662) exactly 10 Cc. of the alcoholic solution, followed by 10 Cc. of potassium iodide T.S., and afterward by 10 Cc. of normal sulphuric acid V.S. When the volume of gas has become constant (within 30 to 60 minutes), note the volume of gas collected. Multiply this volume in Cc. by 0.307, and divide the product by the original weight of the Spirit of Nitrous Ether. At standard temperature and pressure, the quotient will represent the percentage of ethyl nitrite in the liquid. The temperature correction is one-third of one per cent. of the total percentage just found for each degree, additive if temperature is below, subtractive if above, 25° C. (77° F.). The barometric correction is four-thirtieths of one per cent. for each millimeter, additive if above, subtractive if below, 760.

When assayed according to the above method, Spirit of Nitrous Ether should yield not less than 4 per cent. of ethyl nitrite.

Recipe.—Given above.

Tests for Identity.—Given above.

Impurities.—Free acid, excess of aldehyde, details above.

Remarks.—The manufacture of this body, as already outlined on p. 632, is performed by treating alcohol with nitrous acid in the presence of sulphuric acid. Nitrous acid itself is rather an unstable compound, hence in the official preparation it is obtained by treating sodium nitrite with sulphuric acid. Besides, in this and all other esters,

a dehydrating agent (usually sulphuric acid) is necessary to remove the water which is the side product in ester formation (p. 632).

While the process of manufacture of spirit of nitrous ether in the two pharmacopœias (U. S. P. VIII. and U. S. P. 1890) are similar as far as the formation of ethyl nitrite is concerned, the two differ materially as to the separation of the ethyl nitrite from the reacting solution. The recipe of the present pharmacopœia provides that the reaction take place in a vessel surrounded by a freezing mixture, and that the ethyl nitrite formed (insoluble in and lighter than water) be separated from the aqueous layer, freed from acid by treatment with sodium carbonate solution, and from water by use of exsiccated potassium carbonate, and then mixed with twenty-one times its weight of alcohol.

In the process of 1890 the reaction takes place at normal temperatures, and the ethyl nitrite is separated by distillation, the details being as follows:

In a capacious flask is placed sodium nitrite dissolved in water and the alcohol. The flask is tightly fitted with a two-hole cork, through one hole of which passes a separatory funnel, while through the other passes a glass tube connected with an appropriate series of condensers. The sulphuric acid is diluted with water, and while still warm the mixture is allowed to trickle on the sodium nitrite solution in the flask through the separatory funnel.

This causes the following reactions:



The ethyl nitrite liberated, being very volatile, passes over into the condenser, from which it emerges as a light yellow liquid, having the odor of apples, boiling at 18°C .

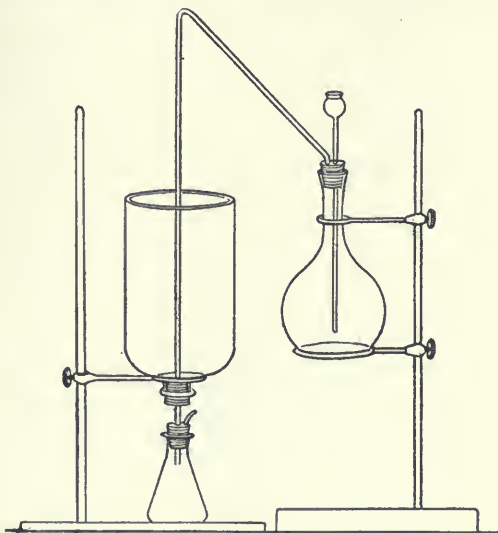


Fig. 222.—Upright condenser for spirit of nitrous ether, U. S. P. 1890.

and with specific gravity 0.900 at 15°C . From this ethyl nitrite traces of acid are removed by washing with a cold solution of sodium carbonate, and the ether is then freed from water by treating with dried potassium carbonate and finally mixed with twenty-one times its own weight of alcohol.

In order to obtain a good yield of the exceedingly volatile ethyl nitrite great

care must be taken for thorough condensation. This is best accomplished by the apparatus shown in Fig. 222, where the vapors are first passed up a tall tube and then passed down in the smaller flask, in transit passing through a layer of ice and salt. By such a method a yield as large as 52 Gm. has been obtained from 55 Cc. alcohol, the theoretic yield being 67 Gm. of nitrite from 55 Cc. alcohol.

The actual operation runs smoothly, provided due care is taken that the ethyl nitrite passes over regularly and without sudden rushes. This is accomplished by constantly agitating the reaction flask. If this is not done, the liquid in the flask forms two layers, the sulphuric acid being below and the sodium nitrite solution above. If these two layers are permitted to form, agitation may result in a sudden production of large quantities of ethyl nitrite with sufficient force to cause an explosion. The temperature of the reaction is usually sufficient to distil over all the ethyl nitrite formed. However, it is a matter of wisdom to finish the operation after the reaction appears to have ceased, by gently warming the mixture by means of a water-bath.

The finished spirit of nitrous ether is an alcoholic solution of pure ethyl nitrite, and does not represent the original *sweet spirit of nitre*, which was prepared by distilling alcohol with a mixture of nitric and sulphuric acids. The alcohol in the latter case acts as a reducing agent on the nitric acid, and, inversely, the nitric acid oxidizes certain portions of alcohol, with formation of aldehyde. The reactions are as follows:



Spirit of nitrous ether contains 4 to 5 per cent. of ethyl nitrite, and as it rapidly deteriorates on standing, should be either freshly prepared or assayed from time to time. The method of assaying is not difficult and quite interesting.

It consists in pouring 5 Cc. of the spirit into an appropriate nitrometer, then adding solution of potassium iodide and diluted sulphuric acid, whereupon the resulting decomposition yields the gas NO or N₂O₂, as shown in the following equation:



The nitrometer is a graduated tube shown in Fig. 223, but in lieu of the expensive apparatus fair results can be obtained by using a Squibb urea apparatus.

The gas, N₂O₂, forces down the salt water with which the nitrometer is filled, and thus the volume of the gas evolved can be easily read. The 5 Cc. of ethyl nitrite should yield 55 Cc. of nitrous oxide gas. The present pharmacopœia makes the assay more accurate by weighing a definite amount of the spirit, diluting with alcohol, and using one-tenth the volume of the dilution. It also directs that the volume of N₂O₂ gas be adjusted to the temperature 25° C., and the barometric pressure of 760 mm. of mercury. (See p. 660.)

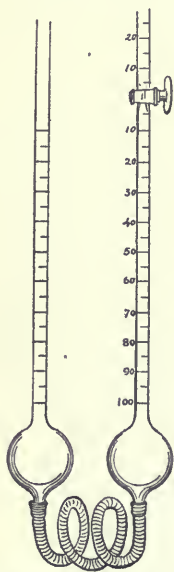
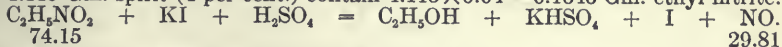


Fig. 223.—Curtman's nitrometer.

The arithmetic of the above statement that 5 Cc. spirit of nitrous ether should yield 55 Cc. nitrous dioxide is as follows:

5 Cc. spirit (sp. gr. 0.823) weigh $5 \times 0.823 = 4.115$ Gm.

4.115 Gm. spirit (4 per cent.) contain $4.115 \times 0.04 = 0.1646$ Gm. ethyl nitrite.



We see from the above equation that

74.15 Gm. ethyl nitrite yield 29.81 Gm. nitrogen dioxide.

Then 0.1646 Gm. ethyl nitrite yields $\frac{74.15}{29.81} \times 0.1646$ Gm. nitrogen dioxide,
or 0.0661 Gm. nitrogen dioxide.

1000 Cc. nitrogen dioxide weigh 1.2272 Gm. (p. 1055).

Then 0.0661 Gm. nitrogen dioxide measures $\frac{1.2272}{1} \times 0.0661$ Cc., or 53.8 Cc.

Hence, it will be seen that a spirit of nitrous ether, 5 Cc. of which will yield 55 Cc. nitrogen dioxide gas, must contain slightly more than 4 per cent. ethyl nitrite.

Sweet spirit of nitre, as the spirit is called, is valuable as a febrifuge and diuretic.

Dose.—2 Cc. (30 minims).

ÆTHER ACETICUS. Acetic Ether.

A liquid composed of about 90 per cent., by weight, of Ethyl Acetate [$\text{CH}_3\text{CO} \cdot \text{OC}_2\text{H}_5 = 87.40$] and about 10 per cent. of alcohol containing a little water. It should be kept in well-stoppered bottles, in a cool and dark place, remote from lights or fire.

A transparent, colorless liquid, of a fragrant and refreshing, slightly acetous odor, and a peculiar acetous and burning taste.

Specific gravity: 0.883 to 0.885 at 25° C. (77° F.).

Soluble in about 7 parts of water at 25° C. (77° F.); miscible, in all proportions, with alcohol, ether, fixed and volatile oils.

Boiling-point: about 72° C. (161.6° F.).

Acetic Ether is readily volatilized, even at a low temperature. It is inflammable, burning with a yellowish flame and an acetous odor.

It should not immediately redden blue litmus-paper.

Upon evaporation, Acetic Ether should leave no residue.

If a portion be allowed to evaporate spontaneously from clean, odorless blotting-paper, the final odor should not resemble that of pineapple (absence of *butylic* and *amylic* derivatives).

When 25 Cc. of Acetic Ether are shaken, in a graduated tube, with 25 Cc. of water just previously saturated with Acetic Ether, upon separation the ethereal layer should not measure less than 22.5 Cc. (absence of an *undue proportion of alcohol or water*).

When a small portion of Acetic Ether is carefully poured upon concentrated sulphuric acid, no dark ring should be developed at the point of contact of the two layers (absence of *readily carbonizable, organic impurities*).

Tests for Identity.—Given above.

Impurities.—Butylic and amylic derivatives; excess of alcohol or water; readily carbonized organic impurities. Details above.

Remarks.—This represents at least 90 per cent. ethyl acetate, the acetic acid ester of ethyl alcohol. The method of manufacture is similar to that of ethyl nitrite, namely, treating alcohol with sodium acetate and sulphuric acid by the following reaction:



The sodium acetate and alcohol are mixed in the distilling flask and the sulphuric acid allowed to trickle in from a separatory funnel, the mixture warmed, and the resulting ethyl acetate distilled.

Acetic ether is a liquid of very delightful odor, and is one of the most popular of the fruit ethers used in preparing artificial flavoring

extracts, the other ethers utilized for the purpose being butyric ether, amyl acetate, and pelargonic ether.

Acetic ether is of considerable value in organic chemistry as a crystallizing solvent. It has but little medicinal use.

Dose.—1 Cc. (15 minims).

Ethyl Sulphate, $(C_2H_5)_2SO_4$.—This is a constituent of the official ethereal oil.

OLEUM ÆTHEREUM. Ethereal Oil.

A volatile liquid consisting of equal volumes of heavy oil of wine and ether.

Recipe.—**Alcohol**, *one thousand cubic centimeters*. 1000 Cc.
Sulphuric Acid, *one thousand cubic centimeters*. 1000 Cc.
Distilled Water, *twenty-five cubic centimeters*. 25 Cc.
Ether, *a sufficient quantity*.

Add the Sulphuric Acid slowly to the Alcohol, mix them thoroughly, and allow the mixture to stand, in a closed flask, for twenty-four hours, or until the liquid is clear; then pour the clear liquid into a tubulated retort of such capacity that the mixture shall nearly fill it. Insert a thermometer through the tubulure, so that the bulb shall be deeply immersed in the liquid, and, having connected the retort with a well-cooled condenser, and also having connected with the receiver a bent glass tube for conducting the uncondensed gases into water, distil, by means of a sand-bath, at a temperature between 150° and 160° C. (302° and 320° F.), until oily drops cease to come over, or until a black froth, which forms on the surface, begins to rise in the retort. Separate the yellow, ethereal liquid from the distillate, and expose it to the air for twenty-four hours, in a shallow dish. Then transfer it to a wet filter, and, when the aqueous portion has drained off, wash the oil which is left on the filter with the Distilled Water, which should be as cold as possible. When this also has been drained off, transfer the oil to a graduated measure, and add to it an equal volume of Ether. Keep the product in small, glass-stoppered vials, in a cool place.

A transparent, nearly colorless, volatile liquid, of a peculiar, aromatic, ethereal odor, and a pungent, refreshing, bitter taste; neutral to dry litmus-paper.

Specific gravity: 0.905 at 25° C. (77° F.).

Remarks.—This product is manufactured by treatment of alcohol with sulphuric acid, allowing the mixture to macerate for twenty-four hours, then pouring same into a retort, and distilling the mixture until the frothy residue of the retort rises to the point where there is danger of it running over. This reaction, which yields varying quantities of ethyl sulphate and ethylsulphovinate, called "*heavy oil of wine*," is dissolved in an equal volume of ether.

In the process large quantities of ether are formed, for it will be noticed that the product is manufactured by a method very similar to that of the manufacture of ether, and with exactly the same constituents, the difference in the manufacture being summed up briefly as follows: Alcohol plus sulphuric acid plus immediate distillation gives ether. Alcohol plus sulphuric acid plus distillation, after standing, gives ethereal oil.

The yield of ethereal oil is about 20 Cc. of the U. S. P. product from 1100 Cc. alcohol, an output so small as to be scarcely worth the pharmacist's time to manufacture. The very fact, however, that the yield is small makes the ethereal oil very expensive, and hence liable to adulteration, and it is said that the larger amount of the so-called ethereal oil on the market consists merely of the last distillate

in the process of the manufacture of ether, and even this is sometimes adulterated, one of the favorable adulterants being castor oil, which liquid has about the same specific gravity as ethereal oil. This falsification, however, can be easily detected by reason of the permanent greasy stain that such ethereal oil will leave on filter-paper, whereas true ethereal oil is uniformly volatile. Ethereal oil is a light yellow liquid, having a peculiar penetrating odor, used as a constituent of compound spirit of ether, and is in itself a stimulant.

The manufacture of ethereal oil gives interesting proof of the presence of carbon in alcohol, the residue from the distillate of a mixture of pure sulphuric acid and alcohol consisting primarily of carbon.

Æthyls Carbamas (U. S. P.).—*Ethyl carbamate* can best be considered among the compounds of carbonic acid (p. 748).

Acetaldehyde is the aldehyde of ethyl alcohol, and its original name has been shortened and employed as the name for the entire class of aldehydes, even as the class alcohols are named after the original type, grain alcohol.

Aldehyde was discovered by Fourcroy and Vauquelin in 1800. Liebig, observing that aldehyde differed from alcohol in the loss of hydrogen atom, named the finished product "alcohol dehydrogenatus." By combining the first syllables of the two words the name aldehyde was devised.

It will be noticed that the aldehydes are not named after the hydrocarbons from which derived, but from the acids they yield on oxidation. Thus the aldehyde under consideration is never called ethyl aldehyde, but acetaldehyde, likewise the aldehyde of methyl is not called methyl aldehyde, but formaldehyde, because eventually yielding formic acid.

Ordinary aldehyde is CH_3CHO ; like other aldehydes, it is made by the oxidation of the corresponding alcohol—in this case, ethyl alcohol. Ethyl alcohol is $\text{C}_2\text{H}_5\text{OH}$, and its formula can be written $\text{CH}_3\text{CH}_2\text{OH}$, or even CH_3CHHOH .

Suppose this be oxidized by bringing it in contact with some substance yielding oxygen, like potassium dichromate and sulphuric acid—we get a reaction like the following, the underscored hydrogen and oxygen atoms separating as water, the new oxygen atom going into the compound:



The manufacture of aldehyde is accomplished by treating ethyl alcohol with a mixture of potassium dichromate and sulphuric acid, a very simple and pretty experiment. The orange-red solution of chromic acid formed when potassium dichromate and sulphuric acid are mixed shows the process of oxidation by changing to green as it parts with its oxygen, the green color being due to the formation of chromium sulphate, $\text{Cr}_2(\text{SO}_4)_3$. Aldehyde is a liquid of characteristic odor, boiling at 21°C .

It is not official, but its polymere, paraldehyde, is accorded pharmacopœial recognition.

PARALDEHYDUM. Paraldehyde.

A polymer of acetaldehyde [$\text{CH}_3\text{COH} = 43.70$]. Paraldehyde should be kept in well-stoppered, dark amber-colored bottles, in a cool place.

A colorless, transparent liquid, having a strong, characteristic, but not unpleasant or pungent odor, and a burning and cooling taste.

Specific gravity: 0.990 at 25°C . (77°F .).

Soluble in 8 parts of water at 25°C . (77°F .) and in 16.5 parts of boiling water; the cold aqueous solution becomes turbid on being boiled. Paraldehyde is miscible, in all proportions, with alcohol, ether, and fixed or volatile oils.

When cooled to near 0°C . (32°F .), Paraldehyde solidifies to a crystalline mass, which becomes liquid again at 10.5°C . (51°F .). It boils at 123° to 125°C . (253.4° to 257°F .), evolving inflammable vapors.

Paraldehyde is neutral or slightly acid to litmus-paper.

When distilled with a small portion of sulphuric acid, Paraldehyde is converted into acetaldehyde, boiling at about 21°C . (69.8°F .). On warming in a test-tube some silver ammonium nitrate T.S., saturated with Paraldehyde, a silver mirror will form on standing.

On heating some Paraldehyde on a water-bath, it should completely volatilize, without leaving any disagreeable odor (absence of *impurities* derived from *fusel oil*).

One Cc. of Paraldehyde should form, with 10 Cc. of water, a clear solution, free from oily drops (absence of *amyl alcohol*, etc.), and portions of this solution, when acidulated with nitric acid, should not be affected by silver nitrate T.S. (absence of *hydrochloric acid*) or barium chloride T.S. (absence of *sulphuric acid*).

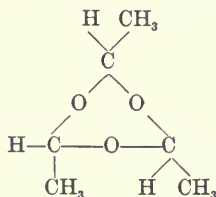
A mixture of 8 Cc. of Paraldehyde and 8 Cc. of alcohol with 1 drop of phenolphthalein T.S. should acquire a pink color upon the addition of 0.5 Cc. of normal potassium hydroxide V.S. (limit of *free acid*).

Tests for Identity.—Given above.

Impurities.—Fusel oil derivative, amyl alcohol, hydrochloric acid, sulphuric acid, excess of free acid. Details above.

Remarks.—This body represents the condensation of three molecules of aldehyde, hence has the formula $(\text{C}_2\text{H}_4\text{O})_3$.

This is the first example of condensation we have considered, and it is of interest to know that such does not merely mean the compression of three molecules of a substance into one molecule of a new compound, but that in this process of changing the character of the compound becomes considerably altered. Thus to paraldehyde is given the structural formula—



The condensation of aldehyde to paraldehyde is accomplished by passing hydrochloric acid gas through the former. Paraldehyde is a liquid of peculiar, pungent odor, specific gravity 0.99, boiling at 123° to 125°C . It is a sedative and hypnotic of particular value in cases of delirium tremens.

Dose.—2 Cc. (30 minims).

CHLORALUM HYDRATUM. Hydrated Chloral.

[CHLORAL, PHARM. 1890]

A crystalline solid, composed of trichloraldehyde or chloral [CCl_3COH] with the elements of one molecule of water. It should be kept in glass-stoppered bottles, in a cool and dark place.

Separate, rhomboidal, colorless, and transparent crystals, having an aromatic, penetrating, and slightly acrid odor, and a bitterish, caustic taste; slowly volatilized when exposed to the air.

Freely soluble in water, alcohol, or ether; also in chloroform, benzene, petroleum benzine, carbon disulphide, fixed and volatile oils. It liquefies when triturated with about an equal quantity of camphor, menthol, thymol, or phenol.

When dried and heated to about 58°C . (136.4°F .), it melts, forming a liquid having a specific gravity of about 1.575, which, at a higher temperature, should not evolve inflammable vapors. Liquefied Hydrated Chloral solidifies to a crystalline mass between 35° and 50°C . (95° and 122°F .).

Hydrated Chloral is decomposed by caustic alkalies, alkaline earths, and ammonia, chloroform and a formate of the base being produced. When warmed with a few drops of aniline and sodium hydroxide T.S., the intensely disagreeable odor of phenyl-isocyanide (isonitril reaction) should be produced.

Hydrated Chloral should be dry, and not readily attract moisture in dry air.

A freshly prepared, aqueous solution of Hydrated Chloral should be neutral to litmus-paper, but it gradually acquires an acid reaction. A neutral alcoholic solution remains neutral permanently.

An aqueous solution of Hydrated Chloral (1 in 20), acidulated with nitric acid, should remain unaffected by silver nitrate T.S. (absence of *hydrochloric acid* and *chlorides*).

If 1 Gm. of Hydrated Chloral be placed in a porcelain dish and be covered with 1 Cc. of nitric acid (sp. gr. 1.38), no yellowish coloration of the mixture should be produced at ordinary temperatures, or even after warming the mixture 3 or 4 minutes, nor should yellowish fumes be produced after ten minutes' warming (absence of *chloral alcoholate*).

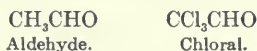
Tests for Identity.—See above.

Impurities.—Hydrochloric acid and chlorides; chloral alcoholate. Details given above.

Remarks.—Chloral hydrate was discovered by Liebig in 1832, but was considered merely as an interesting chemical rarity until its real value as a sedative was discovered in 1869 by Liebreich.

The former pharmacopœial name, *chloral*, is a misnomer, as the true chloral is a liquid which, on addition of one molecule of water, forms the crystalline official substance.

Chloral is an aldehyde, the three hydrogen atoms of whose methyl group are replaced by chlorine, as shown by comparison of formulas:



Chloral is made by passing chlorine gas through alcohol. Chlorine acts as an oxidizing agent—that is, it is one of those substances by which oxygen can be brought in contact with and united to a body. We have seen just above that alcohol, when oxidized, is converted into aldehyde. When chlorine is employed as the oxidizing agent, it also replaces the hydrogen of the aldehyde, forming chloral. The stages of the process are:



The complete equation of the manufacture of chloral from aldehyde is as follows:



In manufacturing chloral by this process there is usually produced, besides chloral hydrate, a body, chloral alcoholate, $\text{CCl}_3\text{CH} < \begin{smallmatrix} \text{OH} \\ \text{OC}_2\text{H}_5 \end{smallmatrix}$, the therapeutic effect of which is quite harmful, and hence great caution should be taken to avoid same in official chloral. The pharmacopœial test for its detection is given above.

Chloral alcoholate is removed during the manufacture of chloral hydrate by upright distillation from concentrated sulphuric acid. This decomposes the alcoholate, while pure chloral is unchanged.

Chloral hydrate is a white solid, occurring in crystals or in white amorphous scales. It is very soluble in water.

As stated above, chloral hydrate is quite soluble in water, and, therefore, it is used to prepare a so-called 100 per cent. solution, that is, a solution of one avoirdupois ounce to a fluidounce. However, it is scarcely worth while to keep the solution on hand, as it can be so easily prepared, and, moreover, the solution spoils on standing, the chloral decomposing into formic and hydrochloric acids.

Chloral hydrate should never be dispensed in alkaline solution, as during the reaction chloroform is produced:



This, as noted on p. 642, is a method of making very pure chloroform.

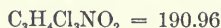
Chloral hydrate is one of the most valuable hypnotics, but, on the other hand, its abuse is one of the most dangerous of the many drug habits.

Dose.—1 Gm. (15 grains).

Chloral hydrate, when rubbed with camphor, thymol, or menthol, is converted into a thick, viscid liquid, the former, under the name *chloral camphor*, has considerable vogue as a local sedative for gum-boils and the like, and, according to the National Formulary, is made by rubbing together equal quantities of chloral and camphor until liquefied.

Quite a number of derivatives of chloral are used as medicines.

CHLORALFORMAMIDUM. Chloralformamide.



A crystalline solid $[\text{CCl}_3\text{CH}(\text{OH})\text{NH}_2\text{COOH}]$, made by the direct union of formamide with anhydrous chloral. It should be kept in amber-colored, well-stoppered vials.

Colorless, lustrous crystals, without odor, and having a somewhat bitter taste.

Soluble in about 18.7 parts of water and in 1.3 parts of alcohol at 25° C. (77° F.). It is readily soluble in ether, glycerin, acetone, and acetic ether. and formamide being produced.

When heated with water to 60° C. (140° F.), it is hydrolyzed, hydrated chloral

When heated to from 114° to 115° C. (237.2° to 239° F.), it melts, but at a higher temperature it is decomposed.

It is not affected by diluted acids, but it is decomposed on warming with alkali hydroxides, the solution becoming at first turbid, and then clear, while chloroform separates.

If 0.2 Gm. be heated carefully in an open dish, it should not give off inflammable vapors, and should volatilize without leaving a weighable residue (absence of *inorganic impurities* and distinction from *chloral alcoholate* and *ethyl carbamate*).

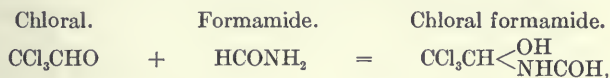
One Gm. of Chloralformamide dissolved in 10 Cc. of alcohol should yield a solution which does not redden moistened blue litmus-paper (absence of *formic, hydrochloric, or other free acids*).

If 1 Gm. of Chloralformamide be dissolved in 10 Cc. of alcohol, the addition of a few drops of silver nitrate T.S. should not at once produce turbidity (absence of *decomposition products*).

Tests for Identity.—Given above.

Impurities.—Inorganic impurities; chloral alcoholate, ethyl carbamate, formic, hydrochloric, or other free acids, decomposition products. Details above.

Remarks.—This new official is made by treating anhydrous chloral with formamide, as expressed in the following equation:



and is typical of a large number of condensation products of chloral

where the group $\text{C} \begin{array}{c} \text{H} \\ \text{—} \\ \text{O} \end{array}$ is converted into the group $\text{C} \begin{array}{c} \text{H} \\ \text{—} \\ \text{OH} \\ \text{—} \\ \text{R} \end{array}$ "R" meaning any radicle with one free bond.

Chloralformamide (or *chloralamide*, as it is sometimes called) is used as a hypnotic.

Dose.—1 Gm. (15 grains).

Ural, or *chloralurethane*, $\text{CCl}_3 \begin{array}{c} \text{OH} \\ \text{—} \\ \text{NH} \end{array} \text{—COOC}_2\text{H}_5$, is made by the combination of chloral and urethane and precipitation of the crystals from the reacting liquid by addition of concentrated hydrochloric acid.

Ural is in the form of transparent crystals melting at 103° C., insoluble in water, and used as a hypnotic.

Hypnal is a mixture of *chloral* (45 per cent.) and *antipyrine* (55 per cent.). It melts at 58° C., and is used as a hypnotic in 1-Gm. doses.

Acetic Acid (CH_3COOH).—This important compound was known to the ancients in the form of vinegar, a product of the fermentation of wine. The concentrated form of acetic acid was not known until 1700, when prepared by Stahl, its production being accomplished only after it was discovered that one of the products of the destructive distillation of wood was acetic acid, this discovery being made by Glauber in 1648, who called the finished product *wood vinegar*.

The chemical analysis of absolute acetic acid was not accomplished until 1814, when Berzelius gave careful study to the structural character of the compound.

Acetic acid occurs naturally in the sap of some plants, is usually a constituent of sweat, and is found as organic esters in some oils; thus, croton oil contains acetin (glyceryl acetate).

Acetic acid has been produced by synthesis from carbon disulphide.

Carbon disulphide plus chloroform gives carbon tetrachloride, CCl_4 .

This body, under treatment with metallic sodium, gives ethylene chloride, $\text{C}_2\text{H}_4\text{Cl}_2$, and eventually ethane hexachloride, C_2Cl_6 . Ethane hexachloride treated with water yields trichloroacetic acid, CCl_3COOH , and this, by reduction, gives acetic acid.

Of course, the above method is of purely theoretic interest, the actual manufacture of acetic acid being either from the distillation of wood or by the oxidation of alcohol.

The latter, when diluted and under the influence of the mother of vinegar, *Mycoderma aceti*, is converted into vinegar, which represents a weak acetic acid. The commercial acetic acid, however, is produced by the destructive distillation of wood.

As already explained on p. 645, wood, on being subjected to heat, yields a tarry distillate, called crude pyroligneous acid (*pyros*, meaning fire, and *lignum*, wood), which is composed chiefly of wood alcohol, acetic acid, and acetone.

The separation of acetic acid from this distillate has already been given, so suffice it to say that a very large quantity of acid is produced by such treatment of wood, and it has been found that the great heat formerly thought necessary to decompose same is unnecessary.

Acetic acid can best be prepared by cutting oak wood in appropriate sized billets, putting into a retort, and heating under 218°C .; at this temperature wood does not char, and except for darkening in color after the process, appears as original wood. The residue is simply dried and is then sold for fuel.

Acetic acid is official in three distinct strengths.

ACIDUM ACETICUM GLACIALE. Glacial Acetic Acid.

A liquid containing not less than 99 per cent., by weight, of absolute Acetic Acid [$\text{CH}_3\text{COOH} = 59.58$] and not more than 1 per cent. of water.

A clear, colorless liquid, of a strong, vinegar-like odor, and a very pungent, acid taste. Specific gravity: not above 1.049 at 25°C . (77°F).

At a temperature somewhat below 15°C . (59°F .) the Acid becomes a crystalline solid. At 117° to 118°C . (242.6° to 244.4°F .) it boils, evolving inflammable vapors.

Glacial Acetic Acid should respond to the tests of purity given under *Acidum Aceticum*; but the tint produced by the addition of 2 drops of tenth-normal potassium permanganate V.S. to 2 Cc. of the Acid diluted with 10 Cc. of water, contained in a clean, glass-stoppered bottle, should not be changed to brown within two hours.

• *Tests for Identity*.—Given above.

Impurities.—See Acetic Acid.

Assay.—Details in Part V.

Remarks.—This glacial acetic acid must contain at least 99 per cent. CH_3COOH , and is called "glacial," because at temperatures below 15°C . it becomes solid, like ice. Some manufacturers have put upon the market an acetic acid labeled "glacial," but on careful examination of the label it is found that in smaller type are the words 80 per cent. That such procedure is dishonest goes without saying. Glacial acetic acid is very valuable as a solvent for a great diversity of substances; thus it dissolves resins, glue, and a large number of other organic bodies. Its use in this way, however, must be with caution, as there is strong likelihood of its forming chemical combinations.

ETHANE DERIVATIVES

ACIDUM ACETICUM. Acetic Acid.

A liquid composed of not less than 36 per cent., by weight, of absolute $[\text{CH}_3\text{COOH} = 59.58]$ and about 64 per cent. of water, and obtained by the oxidation of ethyl alcohol or by the destructive distillation of wood.

A clear, colorless liquid, having a strong, vinegar-like odor, a purely acid taste, and a strongly acid reaction.

Specific gravity: about 1.045 at 25°C (77°F).

Miscible with water or alcohol in all proportions.

When heated, the Acid is volatilized without leaving a residue.

On adding to Acetic Acid enough ammonia water to neutralize it or to leave the Acid in slight excess, and then ferric chloride T.S., the liquid will require a blood-red color, which is discharged by strongly acidulating with sulphuric acid.

When the Acid is slightly supersaturated with ammonia, the liquid should not have a bluish tint (absence of *copper*), nor should any residue be left after evaporating the alkaline liquid on a water-bath (absence of *other fixed impurities*).

If 5 Cc. of the Acid be supersaturated with 10 Cc. of ammonia water, and 5 Cc. of tenth-normal silver nitrate V.S. be added, and the mixture boiled for one or two minutes, no dark deposit should be produced (absence of *formic* or *sulphurous acids*).

When the Acid is slightly supersaturated with potassium hydroxide T.S., the liquid should not develop a smoky odor or taste. If 5 drops of tenth-normal potassium permanganate V.S. be mixed with 2 Cc. of the acid, previously diluted with 10 Cc. of water, and contained in a clean, glass-stoppered vial, the pink tint should not change to brown at once, and should not become entirely brown, or free from pinkish-brown, in less than a half minute (limit of *empyreumatic substances*).

Tests for Impurity.—See above.

Impurities.—Copper, fixed impurities; formic acid, sulphurous acid; excess of empyreumatic substances. Details above. Heavy metals, sulphuric acid, hydrochloric acid. Details in Part V.

Assay.—Details in Part V.

Remarks.—Acetic acid contains 36 per cent. absolute acetic acid, and should be ordered as 36 per cent. acid. The ordinary commercial acetic acid, familiarly called "No. 8," is not the pharmacopœial acid. It contains but 29 to 30 per cent. of absolute acetic acid. The fact that No. 8 acetic acid is not pharmacopœial was learned by a friend of the writer at considerable expense, inasmuch as he dispensed No. 8 acid when acetic acid was called for, and afterward had to answer the charge of violation of the Pure Food Laws of his State. This should warn all pharmacists residing in States having Pure Food and Drug Laws to exercise the utmost caution in dispensing commodities under pharmacopœial names, unless they are exactly the standard prescribed by the pharmacopœia, it being usually considered in interpretation of such laws that when a pharmacist labels a substance with the pharmacopœial name, he means pharmacopœial strength. Thus to dispense essence of vanilla containing tonka bean, labeled "essence of vanilla," would be the ground for legal action.

No. 8 acetic acid is so called because one part of same with eight parts of water is supposed to give the strength of dilute vinegar. *Vinegar* is a 3 to 5 per cent. solution of acetic acid, more or less impure, and it is assumed that it is always made by the fermentation of alcoholic liquids. Thus a dilute white wine or cider, when fermented, will yield vinegar. A quick method for making vinegar is Schutzenbach's method; percolating diluted alcohol over oak shavings. Authorities state that by any of these methods no vinegar containing more than 15 per cent. acetic acid can be made, and as on the market there are a large number of so-called cider vinegars, as strong as 20 to 25 per cent., the conclusion is obvious that such are made from wood vinegar.

The commercial grading of vinegar is by the awkward denomination of so many

"grains," and this means the number of grains of alkali necessary exactly to neutralize one ounce of the vinegar.

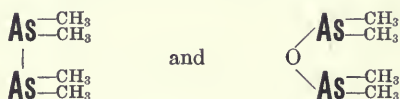
This is even more vague than the average commercial standard, as different authorities say the alkali is sodium carbonate, sodium bicarbonate, potassium carbonate, potassium bicarbonate respectively. Then it is uncertain whether the ounce means troy, avoirdupois, or fluid. If the alkali is potassium bicarbonate, and the "ounce" means fluidounce, "100-grain vinegar" should contain about 12 per cent. acetic acid; 50-grain should contain about 6 per cent. of acetic acid.

Acetic acid is interesting as one liquid of which the specific gravity is not a criterion of strength. Thus at 15° glacial acetic acid has a specific gravity of 1.0562; 85 per cent. has a specific gravity of 1.0747; 81 per cent. is 1.0755; 80 per cent. has a specific gravity of 1.0756, which same specific gravity is possessed by the 79 per cent., 78 per cent., and 77 per cent.; 76 per cent. has a specific gravity of 1.0755, and from that point on a gradual diminution of specific gravity occurs until 43 per cent. has a specific gravity of 1.056—the same specific gravity as the glacial.

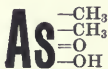
The high specific gravity exhibited by acid containing 77 per cent. to 80 per cent. CH_3COOH is supposed to be due to the formation, in the solution, of hydroxide of acetic acid.

The tests for acetic acid are as follows: When the acid (or an acetate), alcohol, and sulphuric acid are heated together, the mixture gives the pleasant odor of acetic ether. On the other hand, any acetate, when fused with arsenic acid on a platinum foil, gives the disgusting odor of cacodyl.

This substance is really a mixture of *cacodyl*, $\text{As}_2(\text{CH}_3)_4$, and *cacodyl oxide*, $\text{As}_2(\text{CH}_3)_4\text{O}$; the graphic formulas of the two bodies being respectively:



These substances, on treatment with mercuric oxide, yield *cacodylic acid*:



Sodium cacodylate, $(\text{CH}_3)_2\text{AsONaH}_2\text{O}$, the sodium salt of the above, is considerably used as a mild arsenical preparation. Dose, $\frac{1}{2}$ to 2 grains.

ACIDUM ACETICUM DILUTUM. Diluted Acetic Acid.

It should contain not less than 6 per cent., by weight, of absolute Acetic Acid [$\text{CH}_3\text{COOH}=59.58$] and about 94 per cent. of water.

Recipe.—Acetic Acid, one hundred grammes. 100 Gm.
Distilled Water, five hundred grammes. 500 Gm.
To make six hundred grammes. 600 Gm.

Mix them.

Specific gravity: about 1.009 at 25° C. (77° F.).

It should respond to the tests of purity given under *Acidum Aceticum*.

Tests for Identity.—See above.

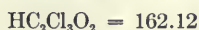
Impurities.—See Acetic Acid.

Assay.—Details in Part V.

Remarks.—Diluted acetic acid, contrary to the general rule for diluted acids (that they be 10 per cent.), contains 6 per cent. absolute acetic acid.

Dose.—2 Cc. (30 minims).

ACIDUM TRICHLORACETICUM. Trichloroacetic Acid.



A monobasic organic acid [CCl_3COOH], usually obtained by the oxidation of hydrated chloral with nitric acid. It should be kept in dark amber-colored, well-stoppered bottles, in a cool place.

White, deliquescent, rhombohedral crystals, having a slight characteristic odor. Very soluble in water, alcohol, and ether. The aqueous solution, on boiling, is decomposed, with the formation of chloroform and carbon dioxide.

Heated to 52°C . (125.6°F .) it melts, and at 195°C . (383°F .) it boils and vaporizes without leaving a residue.

An aqueous solution of Trichloroacetic Acid has an acid reaction upon blue litmus-paper.

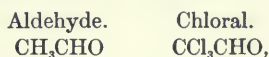
On heating with potassium hydroxide T.S., it is decomposed, with the formation of chloroform and potassium carbonate.

If to its aqueous solution (1 in 10) ferric chloride T.S. be added, a faint reddish color is developed.

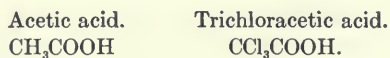
Tests for Identity.—See above.

Assay.—Details in Part V.

Remarks.—As chloral represents aldehyde with three hydrogen atoms replaced by three chlorine atoms,

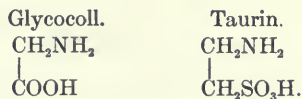


so the trichloroacetic acid represents acetic acid with three hydrogen atoms replaced by three chlorine atoms, viz.:



Again, as acetic acid can be made by the oxidation of aldehyde (see p. 665), so chloral hydrate on oxidation yields trichloroacetic acid. Trichloroacetic acid is used in dentistry for hardening the gums.

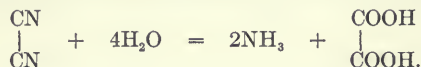
Glycocoll, or amidoacetic acid, and *taurin*, or amidoethylsulphuric acid, must be given passing notice as constituents of oxgall (p. 941). The graphic formulas of these two bodies are:



The *polybasic acids* are derivatives of the hydrocarbons containing more than one carboxyl group. Thus an acid corresponding to the general formula $\text{R}^{\text{ii}} (\text{COOH})_2$, called dibasic acid, and $\text{R}^{\text{iii}} (\text{COOH})_3$, called the tribasic acids, are both illustrations of polybasic acids. The simplest of these is the one which can be considered as derived from ethane.

Oxalic Acid $(\text{COOH})_2$.—This body was long known in the past as the potassium oxalate, which is the constituent of the leaves of several plants, notably *Oxalis perennis*, from which the acid derives its name. It was Scheele, in 1776, who first gave the acid a thorough chemical examination.

Oxalic acid can be made synthetically by several methods. Thus cyanogen, when treated with water, forms ammonia and oxalic acid by the following reaction:



This is stated because it represents the real method of the synthesis of any acid, the same being formed from hydrocarbons containing one less carbon atom than the acid desired by converting same into chloride, then replacing the chlorine group by the cyanogen group (CN), and then converting the cyanogen group into the carboxyl group by the reaction given above, which is considered as a variety of saponification.

The commercial manufacture of oxalic acid is by the oxidation of either sugar or starch by nitric acid, or by fusing cellulose (sawdust) with potassium hydroxide. The latter is the popular method, the yield being 50 per cent. as much as the sawdust employed.

Oxalic acid is not official, although mentioned in the pharmacopœia as a test solution, both in qualitative testing and in volumetric analysis. It is poisonous, and its antidote is any convenient preparation of lime, thus forming insoluble calcium oxalate.

CHAPTER XXXVIII

DERIVATIVES OF PROPANE, BUTANE, AND PENTANE

PROPANE

THE propane derivatives of interest are:

Propyl alcohols.	$\text{C}_3\text{H}_7\text{OH}$.
Propyl aldehyde.	$\text{CH}_3\text{CH}_2\text{CHO}$.
Acetone, U. S. P.	$\text{CH}_3-\text{CO}-\text{CH}_3$.
Sulphonmethane, U. S. P.	$(\text{CH}_3)_2\text{C}(\text{SO}_2\text{C}_2\text{H}_5)_2$.
Sulphonethylmethane, U. S. P.	$\text{C}_2\text{H}_5-\text{C}(\text{SO}_2\text{C}_2\text{H}_5)_2$.
Propionic acid.	$\text{CH}_3\text{CH}_2\text{COOH}$.
Lactic acid, U. S. P.	$\text{CH}_3\text{CHOHCOOH}$.
Glycerin, U. S. P.	$\text{C}_3\text{H}_5(\text{OH})_3$.
Nitroglycerin.	$\text{C}_3\text{H}_5(\text{ONO}_2)_3$. (Official in <i>spirit of glyceryl nitrate</i> .)
Olein.	Glyceryl oleate and other fats.

Of the above, the alcohols have no pharmaceutic interest, it being necessary only to call attention to the fact that two isomeric forms of propyl alcohol are known—the primary and the secondary.

The former yields on oxidation propylic aldehyde, while the secondary gives, under the same treatment, the ketone, acetone. Both of these alcohols yield, with the appropriate acids, esters.

Propionic aldehyde can be dismissed on the simple statement that it, on oxidation, yields propionic acid, $\text{CH}_3\text{CH}_2\text{COOH}$.

ACETONUM. Acetone.

A liquid containing not less than 99 per cent., by weight, of absolute Acetone [Dimethyl-ketone, $\text{CH}_3\text{CO}\cdot\text{CH}_3=57.61$]. It should be kept in well-closed vessels, in a cool place, remote from lights or fire.

A transparent, colorless, mobile, and volatile liquid of a characteristic ethereal odor and a pungent, sweetish taste.

Specific gravity: 0.790 at 25° C. (77° F.).

Miscible with water in all proportions, without cloudiness; also miscible with alcohol, ether, chloroform, and volatile oils.

It volatilizes at low temperatures and boils at 56.5° C. (133.7° F.). It is inflammable and burns with a luminous non-sooty flame.

It should not affect the color of blue or red litmus-paper previously moistened with water.

If 50 Cc. of Acetone be evaporated in a clean glass vessel, no weighable residue should remain.

If 20 Cc. of Acetone contained in a clean glass-stoppered vial be mixed with 0.1 Cc. of tenth-normal potassium permanganate V.S., the pink tint produced by the admixture should not wholly disappear in less than 15 minutes (limit of *empyreumatic substances*).

Tests for Identity.—Given above.

Impurity.—Empyreumatic substances. Details above.

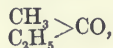
Remarks.—Acetone is the first representative of the ketones (p. 632) to be studied. It has been long known as one of the products of the destructive distillation of wood. It was not, however, until 1832 that its chemical composition became known through the investigations of Liebig.

Acetone has been secured from natural sources (the urine and the blood). It can be made by oxidation of secondary propyl alcohol, as already explained on p. 631. Another chemical method of the manufacture of acetone is by the destructive distillation of calcium acetate by the following reaction:



This method is of importance, inasmuch as it is the type of the general manufacture of ketones.

As seen above, calcium acetate yields dimethyl ketone or acetone. If, instead of calcium acetate, a mixture of calcium acetate and calcium propionate be distilled, we get methyl-ethyl-ketone,



while a mixture of calcium acetate and calcium benzoate gives methyl-phenyl-ketone. The commercial source of acetone, however, is from the destructive distillation of wood, and the acetone is extracted from the distillate, as has already been explained on p. 645.

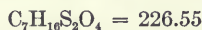
Acetone possesses, in common with all ketones, a number of characteristic reactions; thus it forms crystalline compounds with ammonium bodies, or with potassium bisulphate, and, most striking of all, when treated with hydroxylamine, it forms characteristic bodies called "oximes" (RNOH). It also forms distinct crystalline bodies with phenyl hydrazine, the reagent giving practically similar results with aldehydes. In truth, the two great classes, aldehydes and ketones,

are difficult to distinguish from each other, about the only point of difference being that, while aldehydes oxidize the acids, ketones break up into simpler bodies on oxidation.

The reactions above cited are of pharmaceutical interest only inasmuch as hydroxylamine and phenylhydrazine give distinct crystalline bodies with the sugar (these substances being either of aldehyde or ketone character), hence these reagents are used for testing sugars.

Acetone has but little medical value, its chief use in pharmacy being as a solvent. For this purpose it is used in making the official oleoresins. It yields several products of marked value in medicine, two of which are now official.

SULPHONMETHANUM. Sulphonmethane.



Diethylsulphonedimethylmethane $[(\text{CH}_3)_2\text{C}(\text{SO}_2\text{C}_2\text{H}_5)_2]$, the product of the oxidation of the mercaptol obtained by the condensation of acetone with ethylmercaptan. It should be preserved in well-stoppered vials.

Colorless, inodorous, and nearly tasteless prismatic crystals.

Soluble in 360 parts of water, 47 parts of alcohol, 45 parts of ether, and in 16 parts of chloroform at 25°C . (77°F .); soluble in 15 parts of boiling water and in 2 parts of boiling alcohol; soluble in benzene.

When heated to 125.5°C . (258°F .) it melts, and at a red heat it is consumed, evolving vapors of sulphur dioxide and leaving no weighable residue.

Its aqueous solution is neutral to litmus-paper.

If 0.1 Gm. of Sulphonmethane be heated with 0.1 Gm. of powdered charcoal in a dry test-tube, the characteristic unpleasant odor of mercaptan will be developed.

If 0.1 Gm. be gradually heated with dry sodium acetate, hydrogen sulphide will be evolved.

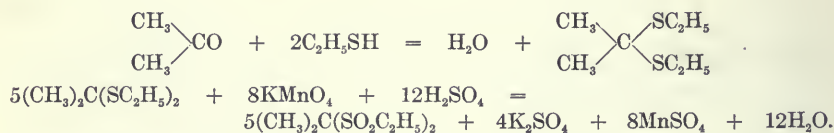
Its solution in boiling water should develop no odor. This solution, after cooling and filtering, should show no turbidity upon the addition of either barium nitrate T.S. or silver nitrate T.S. (absence of *sulphates* and *chlorides*).

If 1 drop of potassium permanganate T.S. be added to an aqueous solution of Sulphonmethane, the liquid should not be immediately decolorized (absence of *readily oxidizable organic impurities*).

Tests for Identity.—See above.

Impurities.—Sulphates, chlorides, oxidizable organic matter. Details above.

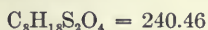
Remarks.—This substance, commonly called *sulphonal*, is made by treating acetone with methyl mercaptan and oxidizing the resulting mercaptid with potassium permanganate, the equation being as follows:



Mercaptan is a liquid of very disagreeable odor, made by treating ethyl bromide with potassium sulphhydrate. It can easily be produced from sulphonal by fusing same in a test-tube with pyrogallol, and this affords a simple test for the identification of sulphonal.

Sulphonal is a white, crystalline solid, sparingly soluble in water, and melting at 125°C . It is a valuable hypnotic.

Dose.—1 Gm. (15 grains).

SULPHONETHYLMETHANUM. Sulphonethylmethane.

Diethylsulphonemethylethylmethane $[(\text{CH}_3)(\text{C}_2\text{H}_5)\text{C}(\text{SO}_2\text{C}_2\text{H}_5)_2]$, a product of the oxidation of the mercaptol obtained by the condensation of methylethylketone with ethylmercaptan. It should be preserved in well-stoppered vials.

Colorless, lustrous, odorless, crystalline scales, which have a bitter taste in aqueous solution.

Soluble in 195 parts of water at 25°C . (77°F .), more readily in boiling water, and readily soluble in alcohol and ether.

When heated to 76°C . (168.8°F .) it melts, and at a red heat is consumed, evolving sulphur dioxide and leaving no weighable residue.

Its aqueous solution is neutral to litmus-paper.

If 0.1 Gm. of Sulphonethylmethane be heated with 0.1 Gm. of powdered charcoal in a dry test-tube, the characteristic unpleasant odor of mercaptan will be developed.

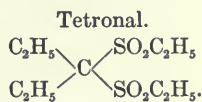
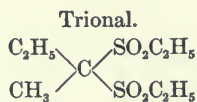
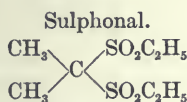
If 0.1 Gm. be gradually heated with dried sodium acetate, hydrogen sulphide will be evolved.

Tests for Identity.—Given above.

Impurities.—Sulphates, chlorides, oxidizable matter. Details as with sulphonmethane.

Remarks.—This body, introduced under the name *trional*, differs from sulphonal by having an ethyl group in place of one of the methyl groups of sulphonal, as is shown below. It thus contains three ethyl groups, hence the name, trional. It is made like sulphonal, except that in place of acetone, ethyl methyl ketone is employed.

Tetronal contains no methyl group, but four ethyls (see compared formula given below), and it is made like sulphonal, only that diethyl ketone is used in its manufacture instead of acetone (dimethyl ketone). Both trional and tetronal are used as hypnotics. *Dose* (of both), 1 Gm. (15 grains).



Propionic acid ($\text{C}_2\text{H}_5\text{COOH}$), discovered by Gottlieb in 1844, can be made by oxidation of primary propionic alcohol, as above stated, and also by the saponification of ethyl nitrile, $\text{CH}_3\text{CH}_2\text{CN}$. It has no pharmaceutic value, but its derivative, oxypropionic acid, is official under the name of **lactic acid**.

Three oxypropionic acids are known: Ethylidene lactic acid, $\text{CH}_3\text{CHOHCOOH}$; ethylene lactic acid, $\text{CH}_2\text{OH}-\text{CH}_2-\text{COOH}$; and sarcosolactic acid. The official acid is the one first mentioned.

ACIDUM LACTICUM. Lactic Acid.

A liquid organic acid, composed of not less than 75 per cent., by weight, of absolute Lactic Acid [$\text{CH}_3\text{CHOH.COOH} = 89.37$] and about 25 per cent. of water.

A colorless, syrupy liquid, odorless, of a purely acid taste, and absorbing moisture on exposure to damp air.

Specific gravity: about 1.206 at 25°C . (77°F .).

Freely miscible with water, alcohol, or ether; insoluble in chloroform, petroleum benzin, or carbon disulphide.

Lactic acid is not vaporized by a heat below 160°C . (320°F .); at a higher temperature it emits inflammable vapors and is finally dissipated; 5 Gm., after combustion, should not leave more than 0.05 Gm. of fixed residue.

Lactic acid has an acid reaction upon blue litmus-paper.

On adding some potassium permanganate to a mixture of equal volumes of lactic and sulphuric acids, and gently heating, the odor of aldehyde will become perceptible.

On adding a few drops of Lactic Acid to 10 Cc. of hot alkaline cupric tartrate V.S., no red precipitate should be formed (absence of *sugars*).

On warming Lactic Acid, the odor of rancid fat should not be noticeable (absence of *butyric* and *other fatty acids*).

If a small portion of the Acid be heated on a water-bath with an excess of zinc carbonate, the mixture dried at 100° C. (212° F.), and then extracted with absolute alcohol, upon evaporation of the latter no sweet residue should remain (absence of *glycerin*).

On carefully pouring Lactic Acid upon an equal volume of colorless, concentrated sulphuric acid contained in a clean test-tube, and keeping the temperature at or below 15° C. (59° F.), no dark-colored zone should develop at the line of contact upon standing for fifteen minutes (absence of more than traces of *organic impurities*).

Tests for Identity.—See above.

Impurities.—Excess of chloride, sulphate, heavy metals. Details in Part V. Butyric and fatty acids, glycerin, organic impurities, and sugars. Details given above. Sarcosine (no change on adding 1 Cc. of copper sulphate test solution to 10 Cc. of solution 1 : 10).

Assay.—Details in Part V.

Remarks.—This body was discovered by Scheele in 1780 as a constituent of sour milk. Besides in milk, it is found in minute traces of opium, and also is found in gastric juice in that form of dyspepsia we call "sour stomach." It can be made synthetically in several ways, but it is of interest merely as establishing the fact that it is a derivative of propionic acid, the commercial source of manufacture being from sour milk.

Milk is a fluid obtained from the cow, and consists of fat (butter), emulsified by means of casein. This emulsion is considerably diluted with water, containing a variety of sugar called sugar of milk.

This sugar of milk, under the influence of certain bacteria found in the air, ferments to lactic acid. The actual method of production from milk is first by removing the butter in churning, then separating the casein by the addition of acid. The liquid remaining after the curd has been removed is called the *whey*, and contains the sugar of milk. If this whey is concentrated and allowed to stand, the milk sugar crystallizes (p. 669), but if the same is allowed to stand until fermentation sets in, lactic acid continues to form until the solution contains 1.6 per cent. acid, when the ferment becomes inactive. If the acidity of the mixture is lessened by treatment with zinc carbonate or chalk, the fermentation continues and the addition of alkali is carried on in actual practice in order to obtain a larger percentage of the acid. Of course, the finished product in such cases is either zinc or calcium lactate, but the acid can be easily freed from the metal by treatment with sulphuric acid. Lactic acid is now being made commercially by fermentation of glucose by the lactic acid ferment.

These details of the manufacture of lactic acid can be applied in therapeutics. As mentioned above, sour stomach is due primarily to the existence of lactic acid produced by methods somewhat similar to those given above. When a certain degree of acidity is reached, the ferment producing this sour stomach becomes inactive; if, on the other hand, there is administered to the patient an alkaline salt, like sodium bicarbonate, the latter may temporarily relieve the pain,

but the very act of neutralizing the free acid gives opportunity for renewed activity of the ferment and the formation of more of the disturbing products, hence the modern treatment of sour stomach is not the use of alkali, but rather the administration of small doses of dilute hydrochloric acid.

Lactic acid is a thick, syrupy liquid, of specific gravity 1.206. It always contains water, the strength of the official acid being but 75 per cent. By removing all the water a new body, *lactone*, is formed.

Dose.—Of lactic acid, 2 Cc. (30 minims).

SYRUPUS CALCII LACTOPHOSPHATIS. Syrup of Calcium Lactophosphate.

This official is made by combining in a mortar 25 Gm. precipitated calcium carbonate with 60 Cc. lactic acid, previously diluted with 100 Cc. water, whereby a solution of calcium lactate is obtained. To this is added 36 Cc. phosphoric acid, diluted with 50 Cc. water, and the mixture triturated until a perfect solution results. After adding 100 Cc. water the liquid is filtered, the mortar rinsed with 50 Cc. water, which is then used to wash the filter. To the mixed filtrate 50 Cc. of orange flower water is added, and 725 Gm. sugar dissolved therein by cold agitation. To the strained syrup enough water is added to make 1000 Cc.

It is used as a nutritive tonic in wasting diseases.

Dose.—8 Cc. (2 fluidrachms).

Polyatomic alcohols are those containing more than one hydroxyl group. Thus while alcohol, C_2H_5OH , containing one hydroxyl, is monatomic, glyecoll, being $C_2H_4(OH)_2$, is diatomic, and glycerin, $C_3H_5(OH)_3$, is triatomic, hence the last two are polyatomic alcohols.

GLYCERINUM. Glycerin. Glycerol.

A liquid obtained by the decomposition of vegetable or animal fats, or fixed oils, and containing not less than 95 per cent. of absolute Glycerol, a triatomic alcohol [$CH_2OH.CHOH.CH_2OH = 91.37$].

A clear, colorless liquid, of a thick, syrupy consistence, smooth to the touch, odorless, sweet to the taste, and producing a sensation of warmth in the mouth; when exposed to the air, it absorbs moisture.

Specific gravity: not less than 1.246 at 25° C. (77° F.).

Soluble in all proportions in water and alcohol; also soluble in a mixture of 3 parts of alcohol and 1 part of ether, but insoluble in ether, chloroform, carbon disulphide, petroleum benzin, benzene, and fixed and volatile oils.

Glycerin is slowly volatilized from weak aqueous solutions, at or above 100° C. (212° F.), with the vapor of water. At boiling temperatures 70 per cent. to 100 per cent. Glycerin rapidly volatilizes; 95 per cent. Glycerin boils at 165° C. (329° F.); anhydrous Glycerin boils at 290° C. (554° F.), without decomposition; under continued heat it is finally entirely decomposed and dissipated.

An aqueous solution of Glycerin is neutral to litmus-paper.

If a fused bead of borax, on a loop of platinum wire, be moistened with Glycerin, and then held in the edge of a non-luminous flame, the latter will be transiently tinted with a vivid green.

Five Cc. of Glycerin, heated to boiling in an open porcelain or platinum dish, and then gently ignited, should vaporize, burn, and leave not more than a dark stain, which on stronger heating should disappear entirely (absence of *mineral impurities*).

If 5 Cc. of Glycerin be mixed with 50 Cc. of water and 10 drops of hydrochloric acid in a small flask, and heated for half an hour on a water-bath, then 10 Cc. of the hot liquid, mixed with 2 Cc. of sodium hydroxide T.S. and 1 Cc. of alkaline cupric

tartrate V.S., should show no yellowish-red cloudiness or precipitate within six hours (absence of *sugars*).

If 5 Cc. of Glycerin be mixed with an equal volume of concentrated sulphuric acid in a test-tube, the liquid should acquire, on standing for one hour, a color not darker than yellow (absence of *readily carbonizable impurities*).

If 5 Cc. of Glycerin be mixed with the same volume of a mixture of equal parts of alcohol and diluted sulphuric acid, and gently heated, a fruity odor should not be recognizable (absence of *butyric acid*).

Tests for Identity.—See above.

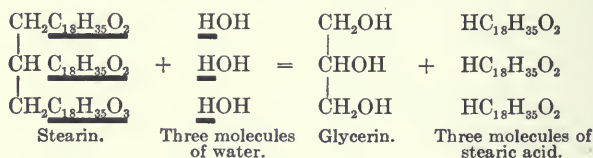
Impurities.—Mineral impurities, carbonizable impurities, butyric acid, and sugars. Details above. Sulphuric acid, oxalic acid, calcium salts, chlorides, heavy metals, and arsenic. Details in Part V. Acrolein (no change on adding ammonium silver nitrate test solution to a 16 per cent. aqueous solution and letting stand five minutes protected from light).

Remarks.—This interesting body was discovered by Scheele in 1789 in the wash-water in making lead-plaster. It was first analyzed by Pelouze in 1837, while its structural formula was worked out by Wurtz (1855). Its introduction as a commercial commodity was between 1844 and 1850, during which time it was introduced by Price, of England, Sarg, of Vienna, and Robert Shoemaker, of Philadelphia.

Glycerin has been made synthetically and by several methods, none of which is of interest save as establishing the structural formula assigned above. It is obtained commercially from fats, these being derivatives of glycerin with oleic, stearic, and palmitic acids (p. 723).

If a fatty oil, such as olive oil, be treated with lead oxide in the manufacture of lead plaster, a double decomposition occurs, with the formation of lead oleate and the freeing of glyceryl hydrate, as fully explained on p. 1133, and this was the original method of the manufacture of glycerin.

In 1854 Tilgman found that glycerin could be obtained from the fats by treating same with superheated steam, and this is the method generally applied at present, the reaction being:



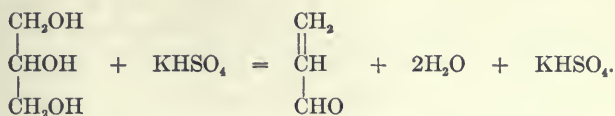
Crude glycerin thus obtained has to be purified by distillation in vacuo, although it is said that the chemically pure product can be distilled without decomposition at ordinary atmospheric pressure.

Glycerin is a transparent, viscid liquid, of sweetish taste, of specific gravity 1.25. As already mentioned, glycerin is a solvent of peculiar value, inasmuch as it is also antiseptic; it is used medicinally as a protective in the form of *carbulated glycerin* (glycerite of phenol, U. S. P.), and when used in the form of glycerin suppositories, it acts as a purgative. Glycerin is also much esteemed as an ingredient of domestic cough remedies.

Dose.—4 Cc. (1 fluidrachm).

Glycerin, when treated with oxalic acid, yields formic acid, and

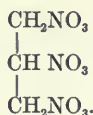
when treated with potassium bisulphate, yields acrolein by the following reaction:



Acrolein is the aldehyde of allyl alcohol, has a very disagreeable odor, and is very irritating to the eyes and throat. It is familiar as the disagreeable odor of burning fat.

Nitroglycerin.—This ester of glycerin was discovered by Sobrero in 1847. It is made by placing glycerin in a freezing mixture and allowing a mixture of nitric acid and sulphuric acid to drip down upon it, cautiously stirring the finished product and then diluting.

Like stearin and olein, nitroglycerin is an ester of glycerin. On p. 723 is given the graphic formula of glycerin and of stearin; to these we will add, for comparison, the graphic formula of nitroglycerin:



Reference to p. 661 will show that the ester, ethyl nitrite, $\text{C}_2\text{H}_5\text{NO}_2$, was made by treating grain alcohol—ethyl hydroxide, $\text{C}_2\text{H}_5\text{OH}$ —with nitrous acid, by the following reaction:



Even so, the ester, nitroglycerin, $\text{C}_3\text{H}_5(\text{NO}_3)_3$, is made by treating the alcohol, glyceryl hydroxide—glycerin, $\text{C}_3\text{H}_5(\text{OH})_3$ —with nitric acid, HNO_3 , by the following reaction:



sulphuric acid being used as the dehydrating agent (p. 661).

Nitroglycerin in concentrated form is used as an explosive, it being one of the most rapid and dangerous of known explosives. As explained on p. 450, an explosive is a body which, under appropriate treatment, will rapidly be converted into a large amount of gas or vapor, and under this test nitroglycerin is one of the most powerful, since one volume of nitroglycerin will, on explosion, yield 750 volumes of gas, while one volume of gunpowder yields only 280 volumes of gas.

Nitroglycerin, while so valuable an explosive, possesses an element of safety, inasmuch as it is not easily set off, it being exploded only by being rapidly heated or by concussion, and in order to obtain the best results the latter method is usually employed, the charge of nitroglycerin being placed in contact with a cap of fulminating mercury, which is fired either by a fuse or by electric connection.

The liquid form of nitroglycerin being inconvenient in many cases, explosive modifications of a solid character have been devised. Thus, *dynamite* is a mixture of three parts of infusorial earth with one part of nitroglycerin.

Nitroglycerin, like all esters, can be saponified by alkali. Thus we have already found that ethyl nitrite, when treated with potassa, is converted into alcohol and potassium nitrite, so in the same way nitroglycerin treated with potassium hydroxide yields inert glycerin and equally harmless potassium nitrate by the following reaction:



This gives a hint as to avoiding explosion if nitroglycerin or its spirit is accidentally spilled.

SPIRITUS GLYCERYLIS NITRATIS. Spirit of Glyceryl Trinitrate. Spirit of Nitroglycerin.

[SPIRITUS GLONOINI, PHARM. 1890]

An alcoholic solution containing 1 per cent., by weight, of Glyceryl Trinitrate [$\text{C}_3\text{H}_5(\text{O}.\text{NO}_2)_3 = 225.44$]. Spirit of Nitroglycerin should be kept and transported in well-stoppered tin cans, and should be stored in a cool place, remote from lights or fire.

Great care should be exercised in dispensing, handling, packing, transporting, and storing the Spirit, since a dangerous explosion may result if any considerable quantity of it be spilled, and the alcohol be partly or wholly lost by evaporation.

If, through accident, it be spilled, a solution of potassium hydroxide should be at once poured over it, to effect decomposition.

A clear, colorless liquid, having the odor and taste of alcohol. *Caution should be exercised in tasting it*, since even a small quantity of it is liable to produce a violent headache. The same effect is produced when it is freely applied to the skin.

It is neutral to litmus-paper.

Specific gravity: 0.814 to 0.820 at 25° C. (77° F.).

On diluting 10 Cc. of the Spirit with 15 Cc. of water—both liquids, as well as the mixture, being brought to 15° C. (59° F.)—the liquid will exhibit at most a faint cloudiness, but the addition of a further portion of 5 Cc. of water should produce a white turbidity.

If the specific gravity of the Spirit be higher than 0.830 at 25° C. (77° F.), or if 10 Cc. of it be rendered turbid by less than 10 Cc. of water, the Spirit should be rejected.

Tests for Identity.—Given above.

Remarks.—This official is a 1 per cent. alcoholic solution of nitroglycerin. It is a colorless liquid, of alcoholic taste. The pharmacopœia, however, gives warning that utmost care must be exercised when testing, as even a small quantity is liable to produce violent headache. It is also well to heed the pharmacopœial warning that the spirit must be handled with the utmost caution, for if spilled, the alcohol will evaporate and the explosive properties of the nitroglycerin will be exerted. Note that the pharmacopœia directs that the danger of explosion is lessened by adding solution of potassa to the spot.

Spirit of nitroglycerin is used as a heart stimulant.

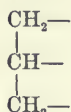
Dose.—0.05 Cc. (1 minim).

Boroglycerin, $\text{C}_3\text{H}_5\text{BO}_3$, is the boric acid ester of glycerin, and is made by heating glycerin with boric acid. For details of operation see Glycerite of Boroglycerin, U. S. P. (p. 207). The official glycerite can be considered as a 50 per cent. solution of boroglycerin, as the following calculation shows:



From the molecular weight just given it is seen that 62 Gm. boric acid plus 92 Gm. glycerin gives 100 Gm. boroglycerin, and as the pharmacopœial recipe (given on p. 207) directs 310 Gm. boric acid plus 460 Gm. glycerin, that this be heated until 500 Gm. remain, and that this be mixed with 500 Gm. glycerin, it is plain that the above quantities (five times the theoretic amounts) will yield 500 Gm. boroglycerin, and that, therefore, the finished glycerite contains 50 per cent. boroglycerin. This is important, inasmuch as boroglycerin is frequently prescribed in 10 per cent., 20 per cent., or 30 per cent. solution, and such should be made by properly diluting the official glycerite with glycerin.

Fats are esters of glycerin with oleic, stearic, and palmitic acids. They will be considered in a special chapter as derivatives of the acids just mentioned. As mentioned above, the radicle—



found in glycerin is called *glyceryl*. This is not to be confused with another radicle (C_3H_5) having the formula $\text{CH}_2=\text{CH}-\text{CH}_2$,—called *allyl*. Allyl is an unsaturated radicle, and, therefore, possesses but one free bond, while glyceryl is a saturated radicle and has three free bonds. Three compounds of allyl are worth passing notice: *acrolein* (allyl aldehyde), $\text{CH}_2=\text{CH}-\text{CHO}$, just described as one of the products obtained on strongly heating glycerin; allyl sulphide, $(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{S}$, the odorous constituent of oil of garlic; and, lastly, allyl isothiocyanate (oil of mustard), which will be considered among the cyanogen derivatives (p. 745).

BUTANE

(C_4H_{10})

The butane derivatives of general importance are:

The butyl alcohols.....	$\text{C}_4\text{H}_9\text{OH}$.
Butyl chloral hydrate.....	$\text{CCl}_3\text{CH}_2\text{CH}_2\text{CHO} + \text{H}_2\text{O}$.
Butyric acids.....	$\text{CH}_3\text{CH}_2\text{CH}_2\text{COOH}$, etc.
Succinic acid.....	$\text{C}_2\text{H}_4(\text{COOH})_2$.
Malic acid.....	$\text{C}_2\text{H}_3(\text{OH})(\text{COOH})_2$.
Tartaric acid, U. S. P.....	$\text{C}_2\text{H}_2(\text{OH})_2(\text{COOH})_2$.

The haloid derivatives of butane are of no pharmacæutic interest. Butyl alcohols, $\text{C}_4\text{H}_9\text{OH}$, are found in four forms, for it will be noticed on p. 635 the number of isomeres increase in proportion to the number of carbon atoms in a compound.

Of these butyl alcohols, the primary $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ is of interest as being one of the constituents of crude fusel oil. On oxidation the primary alcohol yields butyric aldehyde, and then the oxidation product of butyric aldehyde, butyric acid.

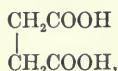
Tertiary butyl alcohol, $(\text{CH}_3)_3\text{COH}$, is of interest because its trichlor-derivative— $(\text{CH}_3)_2\text{CCl}_2\text{COH}$ —is being considerably advertised under the names *chloreton* or *chlorbutanol*. It is made by treating a mixture of chloroform and acetone with caustic alkalis, and is a white crystalline solid, soluble in 125 parts of water, very soluble in alcohol. It is used as a hypnotic in doses of 5 to 20 grains.

A derivative of butyric aldehyde ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CHO}$) is *butyl chloral hydrate* ($\text{CCl}_3\text{CH}_2\text{CH}_2\text{CHO} + \text{H}_2\text{O}$), which was discovered by Kraemer and Pinner in 1873, who, on its discovery, thought it was the isomeric "*croton chloral hydrate*," and under this name the product was introduced into commerce as a hypnotic. *Dose*.—0.3 to 0.6 Gm. (5 to 10 grains).

Two *butyric acids* are known: (1) $\text{CH}_3\text{CH}_2\text{CH}_2\text{COOH}$ (normal).
(2) $(\text{CH}_3)_2\text{CHCOOH}$ (iso).

The first of these is a constituent of sweat, while its glycerin ester comprises 2 to 3 per cent. of butter. From its discovery in butter the name, butyric acid, is obtained.

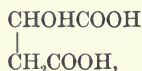
Succinic acid,



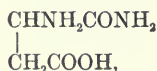
can be considered as butyric acid in which the end methyl group is converted into carboxyl. It occurs naturally in amber (*succinum*), hence its name, and is also found in certain fruit-juices.

Succinic acid is a white, crystalline body, used in medicine to a certain extent for rheumatism and bronchitis. *Dose*.—0.3 to 1.0 Gm. (5 to 15 grains).

Malic acid,

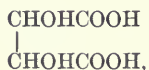


can be considered as monoxysuccinic acid, that is, succinic acid in which one hydrogen is replaced by hydroxyl. Malic acid was discovered in apples, *Pyrus Malus*, by Scheele in 1780, and can be made synthetically from succinic acid. An amide derivative of succinic acid is *asparagin*,



which is found in many plants.

Tartaric acid,



is dioxysuccinic acid, and was found by Scheele in 1789 in cream of tartar. It is interesting as being found in four distinct varieties, and more striking is that all of them seem to have the same graphic formula. The difference among these tartaric acids is chiefly their behavior toward polarized light, the one rotating the ray to the right, hence called dextrogyre, another rotating light to the left, or levogyre; a third does not deviate the ray of light at all, hence called inactive, and the fourth modification, which is also inactive, is racemic acid.

The experiments of the three tartaric acids first mentioned, performed by Pasteur, have become classic, as constituting foundations of the theory of Van t'Hoff ("*Chemistry in Space*").

Pasteur proved that the crystals of the dextrogyre tartaric acid and of the levogyre tartaric acid differ from each other only in the fact that one is the exact mirror

picture of the other. He also proved that the inactive was a mixture of equal quantities of dextrogyre and levogyre tartaric acids.

Van t'Hoff took up the study of the tartaric acids as opening a new field of thought. Up to this time chemical formulas were considered as simply pictures on flat surfaces, while his efforts were directed to attempt to show the exact location of the constituent atoms in a molecule, as to length, breadth, and thickness. It is, of course, far beyond the scope of this work to give any details of this fascinating theory, and the reader is, therefore, referred to Van t'Hoff's book. Suffice it to say that Van t'Hoff's theory is that the real difference among the three tartaric acids is merely the relative difference between the position of the hydrogen, hydroxyl, and carboxyl which they contain, and he gives the following pictures of these three interesting bodies:

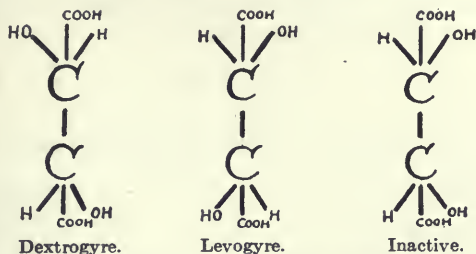
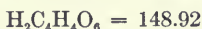


Fig. 224.—The three tartaric acids.

He also discovered a very interesting fact in regard to these bodies, namely, that each bond of the two central carbon atoms of the above pictures is attached to a different atomic grouping; thus one bond is attached to the other central carbon atom, another to a carboxyl, a third to a hydrogen, and a fourth to a hydroxyl. A carbon atom in which each bond is attached to different groups is called an *asymmetric carbon atom*, and is a constituent of all organic bodies which polarize light. This important statement was made about thirty-three years ago, and not only has never been disproved, but is now accepted as a fact. To understand this very striking chapter of newer chemistry thoroughly, a knowledge of polarization is essential, and as this is plainly beyond the limits of the present volume, the reader is advised to study the question in some standard work of physics.

ACIDUM TARTARICUM. Tartaric Acid.



A dibasic organic acid $[\text{C}_2\text{H}_2(\text{OH})_2(\text{COOH})_2]$, usually prepared from argol. It should contain not less than 99.5 per cent. of pure Tartaric Acid.

Colorless, translucent, monoclinic prisms, or crystalline crusts, or a white powder, odorless, having a purely acid taste, and permanent in the air.

Soluble in 0.71 part of water, and 1.67 parts of alcohol at 25° C. (77° F.); in about 0.5 part of boiling water and in about 0.2 part of boiling alcohol; also in 250 parts of ether; nearly insoluble in chloroform, benzene, or petroleum benzin.

When heated for some time at 100° C. (212° F.), the powdered crystals do not suffer a sensible loss of weight. At 135° C. (275° F.) the Acid melts. At a higher temperature it is gradually decomposed, emitting an odor resembling burning sugar, and is finally consumed, leaving not more than 0.05 per cent. of ash.

An aqueous solution of Tartaric Acid reddens blue litmus-paper.

An aqueous solution (1 in 2) of the Acid mixed with a strong solution (1 in 3) of potassium acetate yields a white crystalline precipitate, which is soluble in solutions of alkalis and in mineral acids, but insoluble in acetic acid.

Tests for Identity.—Given above.

Impurities.—Sulphuric acid, oxalic acid, excess of calcium and heavy metals. Details in Part V.

Assay.—Details in Part V.

Remarks.—Tartaric acid occurs in the juice of grapes and in other fruit in the form of acid potassium tartrate and calcium tartrate,

its commercial source being from *argol*, which represents the acid potassium tartrate which deposits from wine during the process of aging. The crude tartar of argol is cooked with chalk, thus forming calcium tartrate, which is then heated with sulphuric acid, forming insoluble calcium sulphate and soluble tartaric acid. A solution of the latter is then evaporated and crystallized.

Tartaric acid is used as a refrigerant, especially in the form of potassium bitartrate. The acid is a constituent of the official Seidlitz powders and is interesting by reason of its ability to form three distinct classes of salts—normal, acid, and double. (See p. 378.)

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

PENTANE

(C_5H_{12})

The pentane derivatives of pharmaceutic interest are:

Amyl alcohols.....	$C_5H_{11}OH$.
Amyl nitrite, U. S. P.....	$C_5H_{11}NO_2$.
Valerianic aldehyde.....	C_4H_9CHO .
Valerianic acids.....	C_4H_9COOH .

The halogen derivatives of pentane are of no pharmaceutic interest. The monatomic alcohols of pentane are not called pentyl alcohols, but go by the name of amyl alcohols, being so called because found abundantly in the spirit obtained by fermenting starch (*amylum*).

The amyl alcohols, $C_5H_{11}OH$, are interesting as being the chief constituents of fusel oil.

There are eight isomeric forms of amyl alcohols possible, and of these, seven are known.

AMYLIS NITRIS. Amyl Nitrite.

[AMYL NITRIS, PHARM. 1890]

A liquid containing about 80 per cent. of Amyl (chiefly Iso-amyl) Nitrite [$C_5H_{11}NO_2$, =116.24], when assayed by the process given below. It should be kept in hermetically sealed glass bulbs or in dark amber-colored, glass-stoppered vials, in a cool and dark place.

A clear, yellowish liquid, of a peculiar, ethereal, fruity odor, and a pungent, aromatic taste.

Specific gravity: 0.865 to 0.875 at $25^\circ C$. ($77^\circ F$).).

Almost insoluble in water; miscible, in all proportions, with alcohol or ether. It is very volatile, even at a low temperature, and is inflammable, burning with a yellow, luminous, and sooty flame. It boils at about 96° to $99^\circ C$. (204.8° to $210.2^\circ F$).).

If 1 Cc. of normal potassium hydroxide V.S. and 10 Cc. of water be mixed in a test-tube with a drop of phenolphthalein T.S., then 5 Cc. of Amyl Nitrite added, and the tube inverted a few times, the red tint of the aqueous layer should still be perceptible (limit of *free acid*).

A mixture of 1.5 Cc. of silver nitrate T.S. and 1.5 Cc. of alcohol with a few drops of ammonia water should not become brown or black if 1 Cc. of Amyl Nitrite be added and the mixture gently heated (absence of *aldehyde*).

Amyl Nitrite should remain transparent, or nearly so, when exposed to the temperature of melting ice (absence of *water*).

Tests for Identity.—Given above.

Impurities.—Excess of free acid, aldehyde, and water. Details above.

Assay.—Details identical with assay for spirit of nitrous ether except that factor is $\frac{\text{volume gas} \times 4.8}{\text{weight of nitrite used.}}$

Remarks.—This ester, which is quite similar to ethyl nitrite (p. 659), is made by practically the same process as the former, namely, by treating amyl alcohol with potassium nitrite and sulphuric acid, and distilling over the reaction products.

It can also be made by treating fusel oil or amyl alcohol with nitric acid and copper, the copper reducing the nitric acid to the nitrous form. This yields the official product, which consists of about 80 per cent. of iso-amyl nitrite, $\text{CH}_3 > \text{CHCH}_2\text{CH}_2\text{NO}_2$, the rest being mixture of nitrites of the other amyl alcohols.

Amyl nitrite is a limpid, light yellow liquid, of pleasant but striking odor, and sharp taste. While it boils at 96° to 99° C., it is very volatile at ordinary temperatures, hence the preservation of amyl nitrite calls for considerable care, and the best form in which it can be put up is in sealed glass tubes rather than in glass-stoppered bottles.

Not merely is loss experienced in amyl nitrite by evaporation, but also by reason of the fact that in presence of moisture it decomposes into alcohol and nitrous acid by the following reaction:



hence, in order to preserve amyl nitrite, it is necessary that it be kept absolutely dry.

Amyl nitrite is a very valuable medicine, because it furnishes a specific in cases of suffocation resulting from attacks of angina pectoris. When sharp spells of this distressing complaint come on, the inhalation of amyl nitrite usually gives relief. Amyl nitrite is put up for this purpose in "pearls," that is, in small hollow shells of thin glass hermetically sealed. When the attack begins, the patient crushes one of these pearls in a handkerchief and inhales the escaping vapor.

The primary amyl alcohol on oxidation yields valerianic aldehyde, and this in turn yields valerianic acid. Secondary amyl alcohols, on the other hand, yield on oxidation either diethyl-ketone, $\text{C}_2\text{H}_5 > \text{CO}$, or methyl propyl ketone, $\text{CH}_3 > \text{C}_3\text{H}_7 > \text{CO}$.

Dose.—0.2 Cc. (3 minims).

Valerianic Acid ($\text{C}_4\text{H}_9\text{COOH}$).—Four modifications of valerianic acid are known:

Propyl acetic acid,	$\text{C}_3\text{H}_7\text{CH}_2\text{COOH.}$
Isopropyl acetic acid,	$\begin{array}{c} \text{CH}_3 \\ \diagdown \\ \text{CH} \\ \diagup \\ \text{CH}_3 \end{array} \text{CH}_2\text{COOH.}$
Methyl ethyl acetic acid,	$\begin{array}{c} \text{CH}_3 \\ \diagdown \\ \text{CH} \\ \diagup \\ \text{C}_2\text{H}_5 \end{array} \text{CH}_2\text{COOH.}$
Trimethyl acetic acid,	$\begin{array}{c} \text{CH}_3 \\ \diagdown \\ \text{C} \\ \diagup \\ \text{CH}_3 \end{array} \text{COOH,}$

and of these, the one usually found in commerce is the isopropyl acetic acid, this body being recognized in pharmacy as *acidum valerianicum*.

Valerianic acid is a constituent of some animal fluids, such as sweat. It is also found in the form of esters in dolphin fat, but the chief natural origin of valerianic acid, from which it obtains its name, was from valerian root, from which it is obtained by distillation with water.

Usually, however, it is made by the oxidation of the appropriate amyl alcohol with potassium dichromate and sulphuric acid.

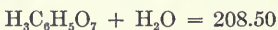
Valerianic acid is a mobile, oily liquid, boiling at 175°C ., of specific gravity 0.935, having the characteristic odor of valerian. It combines with alkalis to form salts, such as the valerianates of ammonium, iron, and zinc, each of which is discussed under its respective metal, hence it is only necessary here to cite that, in order to get a white crystalline sample of ammonium valerianate, monohydrated valerianic acid must be used, and not the trihydrated frequently found in commerce. The trihydrated can be converted into the monohydrated by crystallizing a mixture of neutral sodium valerianate with the crude acid, and treating the crystalline acid salt with concentrated sulphuric acid.

While tricarballic acid, citric acid, and aconitic acid are derivatives of hexane, they can be most conveniently discussed in this chapter, leaving the next chapter for the sole consideration of those very important hexane derivatives, the carbohydrates.

Tricarballic Acid ($\text{C}_3\text{H}_5(\text{COOH})_3$).—This acid with the long name is cited not because of its pharmaceutical value, but merely because it is the connecting link between the compounds just mentioned and of the important organic acid which will be cited next. It will be seen, from the formula given below, that this represents that acid of hexane containing three carboxyls, and which, therefore, may be considered as the oxidation product of primary triatomic hexyl alcohol.

It, therefore, serves the purpose of introducing its oxy-derivative, which is none other than *citric acid*, $\text{C}_3\text{H}_4\text{OH}(\text{COOH})_3$.

ACIDUM CITRICUM. Citric Acid.



A tribasic organic acid [$\text{C}_3\text{H}_4(\text{OH})(\text{COOH})_3 + \text{H}_2\text{O}$], usually prepared from the juice of limes or lemons. It should contain not less than 99.5 per cent. of pure Citric Acid.

Colorless, translucent, right-rhombic prisms; odorless; having an agreeable, purely acid taste; efflorescent in warm air and deliquescent when exposed to moist air.

Soluble in 0.54 part of water and in 1.55 parts of alcohol at 25°C . (77°F .); in about 0.4 part of boiling water and in 1.43 parts of boiling alcohol; also soluble in 18 parts of ether.

When heated to about 75°C . (167°F .) the Acid begins to lose its water of crystallization; at about 135°C . (275°F .) it becomes anhydrous, and melts between 152° and 153°C . (305.6° and 307.4°F .)

When slowly ignited, it is gradually decomposed without emitting an odor resembling burning sugar (difference from *tartaric acid*), and is finally consumed without leaving more than 0.05 per cent. of residue.

An aqueous solution of Citric Acid reddens blue litmus-paper.

On adding 1 Cc. of an aqueous solution of the Acid (1 in 10) to 50 Cc. of calcium hydroxide T.S. (or sufficient of the latter to render the mixture alkaline), the liquid remains clear. Upon boiling this for about one minute, it becomes opaque through the precipitation of calcium citrate, which redissolves on cooling.

If 1 Gm. of the powdered Acid be dissolved in 5 Cc. of a cold solution of potassium acetate (1 in 3), the liquid should remain clear, even after the addition of an equal volume of alcohol (absence of *tartaric* or *oxalic acid*).

Tests for Identity.—See above.

Impurities.—Tartaric acid and oxalic acid. Details above. Calcium, heavy metals, excess of iron, excess of sulphuric acid. Details in Part V.

Assay.—Details in Part V.

Remarks.—Citric acid was isolated by Scheele in 1789 from the juice of lemon, and analyzed by Liebig in 1831; while the structure of the body was proposed by Salet (1868) and confirmed by Grimaux and Adam (1880).

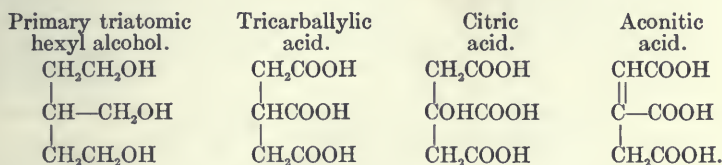
Citric acid occurs in the juice of the lemon and some other fruits, the former, however, being the source of the commercial article. By treating lemon-juice with calcium carbonate, calcium citrate is precipitated out, and this precipitate, after being thoroughly washed, is treated with sulphuric acid, whereby a precipitate of calcium sulphate is formed and citric acid goes into solution. The citric acid solution is then concentrated in vacuo and allowed to crystallize.

Several syntheses of citric acid have been accomplished, one of which is from carballylic acid, thus proving its structural formula. None, however, has attained commercial value.

Citric acid occurs in translucent, right-rhombic prisms, melting at 152° to 153° C., having the specific gravity 1.6, and soluble in 0.63 part of water.

Citric acid crystallizes with one molecule of water of crystallization, and is prone to efflorescence. Moreover, if the acid is heated to 175° C., it will not merely lose its water of crystallization, but also a molecule of water from its own structural formula, with formation of a new body, *aconitic acid*.

It may be well to compare the graphic formula of the three acids just mentioned along with the alcohol from which derived:



The fact that citric acid easily parts with a molecule of water should always be borne in mind, and, therefore, undue heat should not be used in evaporating a solution of citric acid or of its salts. In fact, the evaporation should take place in vacuo.

Dose.—500 milligrammes (7½ grains).

Citric acid forms, with metals, the well-known salts, the citrates. Of these, the following are official:

Bismuth citrate.
Bismuth and ammonium citrate.
Ferric citrate.

Iron and ammonium citrate.
Iron and quinine citrate.
Soluble iron and quinine citrate.
Iron and strychnine citrate.
Soluble iron and strychnine citrate.

Lithium citrate.
Effervescent lithium citrate.
Effervescent magnesium citrate (U. S. P. 1890).
Potassium citrate.
Effervescent potassium citrate.

DRUGS CONTAINING FRUIT ACIDS

As this opens the subject of crude drugs, a few words of introduction may be advisable.

Vegetable drugs consist of whole plants, plant parts, or plant excretions; in other words, are portions of that collection of cells called a plant. Knowledge of plants forms the science of botany, and a full discussion of this science is clearly beyond the limits of this work; hence the student is referred to list of standard text-books given in the Preface. To explain the "official definitions" (p. 24) of the drugs that must be considered in this book a slight résumé of the important plant parts is in order and is here given, fuller details of peculiar cases being considered under drugs illustrative of each case.

The study of vegetable drugs—of the plant parts used in medicine—forms that branch of knowledge called organic materia medica, and even this in its complete form is beyond the province of this work, reference to the leading text-books on the subject being found in the Preface.

In the study of materia medica, drugs are usually grouped as belonging to the several classes of plant parts,—roots, stems, rhizomes, tubers, woods, barks, leaves, bulbs, herbs, flowers, fruit, and seeds,—hence it may be well to define these parts (or organs) at this place.

A *root* is that part of the plant axis that does not bear leaves. Roots are not always under ground; thus, ivy has roots above ground that aid the plant in climbing.

A *stem* is that portion of the plant axis which does bear leaves. All stems are not above ground; the so-called "roots" of the violet or of Solomon's seal, which run horizontally under ground, are stem parts, for the leaves spring directly from them.

A *rhizome* is that stem part which lives under ground. The violet stem part just mentioned is an example of a rhizome.

A *tuber* is a thickened underground stem, used as a reservoir for starch. Example, the Irish potato. In the case of some tubers, they more closely resemble roots than stems, and are then called *tuberous roots*; an official example of such is found in aconite.

Stems of large plants like trees are called *trunks*, and such a *trunk* can be roughly divided into the central part, called the *wood*, and the outer, more or less corky layer, called the *bark*.

Leaves are expansions of the stem tissue arranged regularly on the stem. Ordinary leaves perform the function of respiration or breathing, playing an important part in the conversion of carbon dioxide, which they absorb from the air, into starch, sugar, and cellulose.

Bulbs are collections of underground leaves attached to a stem which consists of nothing more than a conical disk from beneath which the rootlets project. The onion is a good example of a bulb. The difference between a tuber and a bulb may well be given here. An example of a tuber is the Irish potato, and as the example of a bulb the onion will be taken, and even slight examination will show that these two are different. Both grow underground and neither is a root; yet they are not the same. A bulb, like a tuber, is an underground stem, but it goes farther, since it also includes distinct underground leaves. Thus, in the onion, the thick, juicy scales are the underground leaves,

and all there is of the stem is the hard little base from which the roots grow and to which the scales (leaves) are attached.

Herbs represent the whole dried plant, usually deprived of its roots.

Flowers are leaf modifications which perform the function of reproduction, the special organs for this purpose being the ovary—the female organ—and the anther—the male organ. The fertilized ovary develops within itself—

Seeds, which are organisms containing a germ,—the beginning of a new plant,—provided with sufficient nourishment to support it during the first days of its sprouting.

Fruits represent the ripened ovary and its appendages. The appendages vary from a thin, chaff-like ovary wall, in anise, to the large luscious “meat” and thick green rind of the watermelon.

Chemically, drugs consist of many substances; those that are plant organs consisting, histologically, of cell-wall and cell-contents, drugs that are exudations from plants being usually entirely cell-contents. This cell-wall, which consists of cellulose, wood, bast, or cork, according to its development is generally spoken of pharmaceutically as “*fibrous tissue*.” The cell-contents consist of a vast number of bodies, such as alkaloids, glucosides, neutral principles, oils (volatile or fixed), tannin, resin, gum, starch, sugar, etc. The last four are found in almost all plants, while the others are more sparingly found, and constitute the active principle of the specific drug under consideration.

This leads to the classification of drugs into alkaloidal drugs, glucosidal drugs, astringent drugs, etc. In the consideration of every drug on the pages which follow the *constituents* will be an important feature study, and let it be here frankly stated that under constituents will be given only those which make the drug of value. Thus in considering alkaloidal drugs a continuous repetition of “gum, starch, and sugar” will be omitted, reference being made to these constituents only when they play an important rôle.

RHUS GLABRA. *Rhus Glabra*.

The dried fruit of *Rhus glabra* Linné (Fam. *Anacardiaceæ*).

Constituents.—Potassium and calcium malate; some tannin.

Medical Properties.—Refrigerant, astringent. Infusion used as a gargle in sore throat.

Dose.—1 Gm. (15 grains).

TAMARINDUS. *Tamarind*.

The preserved pulp of the fruit of *Tamarindus indica* Linné (Fam. *Leguminosæ*).

Constituents.—Citric acid, tartaric acid, malic acid, and potassium bitartrate.

Remarks.—Tamarind is a preserved pulp of the fruit rather than the whole fruit, and is frequently found in commerce mixed with molasses.

Medical Properties.—Refrigerant and laxative. A drink from tamarinds and cream of tartar is popular in the south for “thinning the blood.”

Dose.—16 Gm. (240 grains).

LIMONIS SUCCUS. Lemon Juice.

The freshly expressed juice of the ripe fruit of *Citrus Limonum* Risso (Fam. *Rutaceæ*).

A slight turbid, yellowish liquid, having the odor of lemon; taste acid and often slightly bitter.

Specific gravity: 1.030 to 1.040 at 25° C. (77° F.).

It reddens blue litmus-paper and should contain from 7 to 9 per cent. of citric acid.

Tests for Identity.—Given above.

Impurities.—Sulphuric acid or sulphates, acetic acid, tartaric acid.

Details in Part V.

Assay.—Details in Part V.

Constituents.—Citric acid, sugar, some salts ($\frac{4}{10}$ of 1 per cent. ash).

Remarks.—As mentioned on p. 689, lemon juice is the commercial source of citric acid. Note that the original "neutral mixture" (Mistura Potassii Citratis, p. 444) was made by neutralizing lemon juice with potassium bicarbonate.

Medical Properties.—Refrigerant, formerly used on shipboard as a preventive of scurvy, a loathsome disease produced by a steady diet of salt meat.

Dose.—30 Cc. (1 fluidounce).

CHAPTER XXXIX**DERIVATIVES OF HEXANE**

THE following derivatives of hexane (C_6H_{14}) are of interest:

Hexyl alcohols.....	$C_6H_{13}OH$.
Capronic acid.....	$C_5H_{11}COOH$.
Mannite }	$C_6H_8(OH)_6$.
Dulcitol }	
Sorbit }	
Glucose.....	$C_6H_{12}O_6$.
Sugar, U. S. P.....	$C_{12}H_{22}O_{11}$.
Starch, U. S. P.....	$(C_6H_{10}O_5)_x$.
Gum.....	$C_6H_{10}O_5$.
Cellulose.....	$(C_6H_{10}O_5)_x$.
Pyroxylin, U. S. P.....	Nitrocellulose.
Glucosides.....	
Tricarballic acid.....	$C_3H_5(COOH)_3$.
Citric acid, U. S. P.....	$C_3H_4(OH)(COOH)_3$.

Of the *hexyl alcohols*, seventeen are possible, but since none is of pharmaceutic value, they will not be noticed.

By the oxidation of one of these alcohols, *capronic acid*, $CH_3(CH_2)_4COOH$, is obtained. This capronic acid is a fatty acid discovered by Chevreul in 1824, and is found as an ester in cocoanut oil, Limburger cheese, and goat butter, and is cited merely because it is the only monobasic acid of hexane of pharmaceutic interest.

Mannite, $C_6H_8(OH)_6$, is the hexatomic alcohol of hexane. It was isolated by Proust from manna in 1800, and is also found in minute traces in *Viburnum Opulus* and in a few other barks. The chief source

of commercial mannite is manna, from which it is obtained by treating with boiling alcohol. From the alcoholic solution the mannite separates on cooling in small white crystals of sweetish taste.

It should be called *mannitol*, since it is an alcohol.

By careful oxidation it yields *mannose*, a variety of sugar, while oxidation with nitric acid yields *saccharic acid*.

Dulcite and *sorbite* are isomeres of mannite, the former obtained by reduction of lactose (milk-sugar) and the latter by reduction of glucose.

These three hexatomic alcohols yield ketone and aldehyde derivatives, which are of great importance, such as *glucose*, *mannose*, *cane-sugar*, *starch*, and *cellulose*.

The formula of the first two is $C_6H_{12}O_6$.

Cane-sugar is $C_{12}H_{22}O_{11}$.

Starch and cellulose, $(C_6H_{10}O_5)_x$.

On examining these formulas it will be noticed that the hydrogen and oxygen atoms attached to the carbon atoms are present in exactly the same proportion as in water, viz., there are always twice as many hydrogen atoms as oxygen atoms. For this reason to this class of bodies was assigned the name "*carbohydrates*," although at present but little stress is laid on this characteristic.

Carbohydrates.—While most carbohydrates are derivatives of hexane, some are found containing more or less carbon than six. Thus, arabinose is $C_5H_{10}O_5$. On the other hand, some contain seven carbon atoms, some eight carbon atoms, and some nine; but practically all the carbohydrates are derivatives of hexane.

The hexane carbohydrates can be roughly grouped into three classes, the first having the form $C_6H_{12}O_6$, which have representatives in glucose and dextrose; the second, being of double formula, $C_{12}H_{22}O_{11}$, is found in cane-sugar; while the group $C_6H_{10}O_5$ has representatives in such well-known bodies as starch, gum, and cellulose.

The first examination of these bodies shows that one should be derived from the other, thus:

$$\begin{array}{rcl}
 2C_6H_{10}O_5, \text{ which is} & & C_{12}H_{20}O_{10} \\
 \text{plus one molecule of water,} & & \underline{H_2O} \\
 \text{gives us a molecule of sugar} & & C_{12}H_{22}O_{11}, \\
 \text{and when to this we add another} & & \\
 \text{molecule of water,} & & \underline{H_2O} \\
 \text{we get} & & C_{12}H_{24}O_{12} \\
 \text{or} & & \underline{2C_6H_{12}O_6} \\
 \text{which is the formula of glucose.} & &
 \end{array}$$

The transition of starch or sugar into glucose is really performed by the adding of water (by chemical means). The reverse operation is not so simple a one, and has not been performed.

On the other hand, one molecule of glucose minus one molecule of water gives the empiric formula of one molecule starch or gum.

As in the case cited above, the transformation of glucose into starch has not been accomplished, but the reverse operation, the conversion of starch into glucose, is performed by the very simple act of heating the starch with dilute sulphuric acid. It may be as well to state right here that the agent used for the molecular addition of water is usually diluted sulphuric acid, and the process of chemically combining water is called "*hydrolyzing*." On the other hand, if it is desired to remove

water from the molecule of an organic substance, concentrated sulphuric acid is usually the agent employed, when it is said to act as a "dehydrator."

The formula, $C_6H_{12}O_6$, it will be noticed, differs from the formula of mannite, $C_6H_{14}O_6$, by containing two atoms of hydrogen less. It will be also noticed that this is the difference between the formula of an ordinary alcohol and its aldehyde or ketone, and it is really true that $C_6H_{12}O_6$ is an aldehyde or ketone derivative of the hexatomic alcohol, $C_6H_8(OH)_6$. The carbohydrates are called *aldoses*, or *ketoses*, according as the alcohol group has been changed to an aldehyde or a ketone, though some are of more intricate composition.

The carbohydrates can best be studied by taking up three groups in their regular order, viz.:

Those having formula $C_6H_{12}O_6$, or monoses.

$C_{12}H_{22}O_{11}$, or bioses.

$C_6H_{10}O_5$, or polysaccharides.

THE CARBOHYDRATES

($C_6H_{12}O_6$)

These are bodies of more or less sweetness, and all possess optical activity, that is, all deviate the ray of polarized light either to right or left, except the synthetic inactive varieties. According to Van t'Hoff's rule, each active substance should contain at least one asymmetric carbon atom, and investigation has shown that this is true in every case. Several syntheses of these bodies have been accomplished; thus paraformaldehyde cooked with milk of lime gives acrose; formaldehyde cooked with milk of lime gives a mixture of formose and acrose (p. 647). Likewise dibromide of acrolein (p. 681), when treated with barium hydroxide, gives acrose. The bodies of this series are all readily converted into alcohol when brought into contact with the ferment, yeast. If other bacteria of fermentation are added, they yield lactic or butyric acid or dextrin, as the case may be.

Following the rule of aldehydes and alcohols, these bodies of formula $C_6H_{12}O_6$ oxidize to special acids under special treatment, and, on the other hand, can be reduced to special alcohols.

They all combine with calcium or strontium to form alcoholates of the same. They give all the reactions characteristic of aldehydes and ketones, for example, form distinct crystalline bodies with phenyl hydrazine. Some bodies of the group (mannose and glucose) oxidize to the corresponding acid showing aldehyde characteristics, while the fructoses, under similar treatment, break up, showing their ketone characteristics.

The bodies of formula $C_6H_{12}O_6$ can be roughly divided into three groups:

Fructoses, $CH_2OH-(CHOH)_5CO-CH_2OH$.

Mannoses, $CH_2OH-(CHOH)_4-CHO$.

Glucoses, $CH_2OH-(CHOH)_4-CHO$.

Of each of these, three varieties are known: (1) The levogyre, which rotate the ray of light to the left; (2) the dextrogyre, which rotate the ray to the right; and (3) the inactive, which do not affect the ray of light.

It will be noticed that levogyre is abbreviated to the letter "l," dextrogyre, to "d," and inactive, to "i," and let it be stated here that in future, in this work, these abbreviations will be used instead of the long technical names.

The three fructoses are as follows:

"l" fructose (*acrose*), which is of interest merely as one of the synthetic sugars.

"d" fructose (*fruit-sugar*), which is the sweet constituent of many fruits.

"i" fructose—*levulose*.

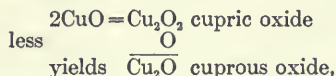
The three mannoses are of no pharmaceutic interest except as derivatives of mannite.

Of the three *glucoses*, the only one of interest is the dextrogyrate.

This product is the chief constituent of honey (which see), and can be made by inversion (hydrolysis) of cane-sugar and of starch, the

reaction being as given on p. 693. The commercial source of glucose is corn, from which the embryo is removed by mechanical means, the residue treated with sodium hydroxide to remove gluten and albuminous matter naturally found in the corn, and the starch thus purified hydrolyzed by treating with sulphuric acid. On completion of the inversion, the excess of acid is neutralized by the addition of barium chloride, the precipitate of barium sulphate filtered out, and the aqueous solution of glucose concentrated in vacuo. It is interesting to note that the separated embryo of the corn, when submitted to expression, yields a considerable quantity of fixed oil (*corn oil*), which is a valuable drying oil. The original intent of removing the germ was to prevent the glucose liquid from being greasy, and this affords another instance of the development of a special industry by the utilization of side-products. Glucose is placed on the market in two forms: one, a thick, syrupy liquid, having specific gravity 1.412, perfectly miscible with alcohol and water.

A solid form of glucose is now obtainable, the terms *dextrose* and *grape-sugar* being at present reserved for this solid glucose. Glucose is a reducing agent, changing cupric salts to the cuprous form. Just as sulphuric oxide, SO_3 , can be converted into sulphurous oxide, SO_2 , by reduction, so can cupric oxide, CuO , or its hydroxide, dissolved in excess of alkali, be reduced to cuprous oxide, Cu_2O , as shown below:



the valence of the copper being reduced during the reaction from II to I.

While this reaction occurs under the influence of any reducing agent, it is usually confined to the identification and estimation of glucose, for which it is admirably adapted, the brick-red precipitate of cuprous oxide being very characteristic. Glucose is a constituent of urine in diabetes, and in this way its identification is of much importance to the pharmacist. In examining the urine for sugar either Fehling's or Trommer's test is applied. The latter, which is the oldest, consists in treating the suspected urine with a solution of copper sulphate and a few drops of solution of soda (whereby the copper sulphate is converted into cupric hydroxide).

On boiling, this blue cupric hydroxide is converted into brick-red cuprous oxide if glucose or other reducing agent be present.

Fehling's solution is an improved modification intended for volumetric analysis. It contains sodium and potassium tartrate (Rochelle salt), in addition to copper sulphate and sodium hydroxide. It is found among the volumetric solutions of the pharmacopœia. (See Part V.)

Galactose ($\text{CH}_2\text{OH}(\text{CHOH})_4\text{CHO}$).—This monose is made by inversion of milk-sugar. It oxidizes to mucic acid, and is of no pharmaceutic value.

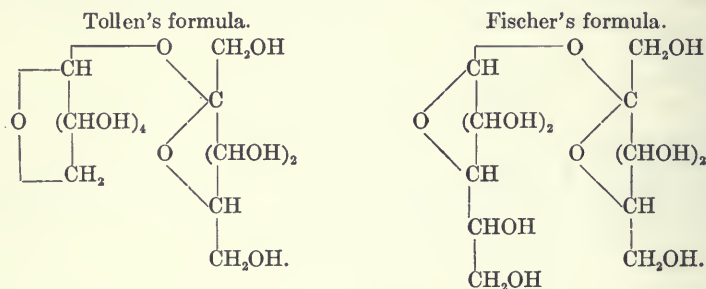
CARBOHYDRATES



The **bioses** are those carbohydrates having the formula $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, and, as mentioned above, these represent two molecules of $\text{C}_6\text{H}_{12}\text{O}_6$ minus one molecule of water.

They are much more easily crystallized than the monoses, are optically active, and are fermentable only after being hydrolyzed, the sole exception to this statement being that maltose is directly fermentable. As already mentioned, the bioses, under treatment with dilute sulphuric acid and special ferments, such as diastase, are hydrolyzed into bodies having the formula $C_6H_{12}O_6$. Each biose yields different products under hydrolysis; thus cane-sugar hydrolyzes to glucose and fructose; maltose to glucose; and lactose into a galactose and glucose.

Maltose and lactose reduce silver nitrate and Fehling's solution, hence are aldehydes. Cane-sugar, on the other hand, does not act in this way, hence contains no aldehyde groups. All three of these bioses form esters of acetic acid containing eight acetyl groups, from which is deduced the fact that each contains eight hydroxyls. There is much in the chemistry of these bodies yet to be cleared, but from the few facts already mentioned it has been decided that they represent anhydrides of the various glucoses, evidently those into which they separate under hydrolysis. The exact chemical structure of these bodies has not been clearly ascertained, but the following formulas have been suggested for saccharose (cane-sugar):



SACCHARUM. Sugar.

$$C_{12}H_{22}O_{11} = 339.6$$

The refined sugar obtained from *Saccharum officinarum* Linné, and from various species or varieties of *Sorghum* (Fam. *Gramineæ*); also from one or more varieties of *Beta vulgaris* Linné (Fam. *Chenopodiaceæ*).

White, dry, hard, distinctly crystalline granules, odorless, and having a purely sweet taste. Permanent in the air.

The aqueous solution, saturated at 25° C. (77° F.), has a specific gravity of about 1.340, is miscible with water in all proportions, should be colorless, and is dextrogyrate.

Soluble in 0.46 part of water and in 137.2 parts of alcohol at 25° C. (77° F.); in 0.2 part of boiling water and in 28 parts of boiling alcohol; also soluble in 80 parts of boiling absolute alcohol, but insoluble in ether, chloroform, or carbon disulphide.

The aqueous or alcoholic solution of Sugar is neutral to litmus-paper.

Both the aqueous and the alcoholic solution of Sugar should be clear and transparent. When kept in large, well-closed and completely filled bottles, the solutions should not deposit a sediment on prolonged standing (absence of *insoluble salts*, *ultramarine*, *Prussian blue*, etc.).

If 1 Gm. of Sugar be dissolved in 10 Cc. of boiling water, the solution mixed with 4 or 5 drops of silver nitrate T.S., then about 2 Cc. of ammonia water added, and the liquid quickly brought to the boiling-point, not more than a slight coloration and no black precipitate should appear in the liquid after standing at rest for five minutes (absence of *glucose*, and of more than a slight amount of *inverted sugar*).

Tests for Identity.—Given above.

Impurities.—Insoluble salts, ultramarine, Prussian blue, glucose, and inverted sugar. Details above.

Remarks.—Sugar can be obtained from cane, *Saccharum officinarum*, beet, *Beta vulgaris*, or sorghum, *Andropogon Sorghum*, var. *Saccharatus*, the products in each case being chemically identical. The

three varieties of commercial sugar are yielded by three distinct plants; those yielding sugar-cane and sorghum sugar greatly resemble the corn, and in both cases the stalks are cut near the roots at the proper season, the leaves lopped off from the stalks, which are then passed through rollers, by which means the juice is expressed. The juice is treated with lime to remove the gluten and albumin, and the lime removed as calcium carbonate, when the liquid consists of a thin solution from which sugar is obtained by appropriate concentration. At present, in place of lime, calcium bisulphite is frequently used. This chemical not merely removes the gluten and albumin, but also acts as an antiseptic. Formerly the light juice was concentrated in large pans under atmospheric pressure ("open kettle process"). In such cases, however, the solution underwent slight decomposition, darkening the product, and forming the commercial brown sugar. To refine this brown sugar it is necessary to redissolve and clarify it with albuminous matter, such as blood, filter through charcoal and the solution concentrated in vacuo, and at the present time this concentration in vacuo is done from the very beginning. When the liquid in the vacuum pan has reached a proper degree of concentration, and the sugar is beginning to separate out by crystallization, the mass is transferred to centrifugal machines and is entirely freed from moisture.

The process of making beet-sugar is more complicated than that of making cane-sugar, the saccharine solution being prepared by chopping the beets into small particles and macerating same in warm water, this process being known as the diffusion process. This solution contains many more impurities than does the cane-juice, hence many and complicated methods of purification have to be used, and in this way quite a number of side-products of more or less value are obtained. Thus, it will be recalled that methyl chloride (p. 641) is now obtained largely from beet-sugar residue.

The process of diffusion mentioned above is now used in some factories in the manufacture of cane-sugar. The cane, instead of being run through rollers, is relieved of its sugar by maceration in warm water. The extraction in this case is much more thorough than by pressure, but requires more manipulation, making the process more expensive.

Sugar appears in commerce usually in the granulated form, although in some sections of the world the old-fashioned sugar-loaf is still used. This is made by pouring the most concentrated warm solution of sugar into conical molds, letting stand twenty-four hours, draining off the syrup, and drying.

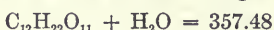
If a saccharine solution is allowed slowly to crystallize on strings, large transparent crystals are formed, these being known as *rock-candy*. Sugar melts at 160°C ., and if the sugar which has been brought to the melting-point with the aid of a little water is then allowed suddenly to cool, it produces a candy form of sugar sometimes called *barley-sugar*. If sugar is heated to 200°C ., the product begins to carbonize and results in a deep brown liquid called *caramel*. This liquid is used quite largely as a coloring matter in soda-water syrups; hence it will not be amiss to mention the details of preparation. Thus, if the carameled sugar is allowed to cool without addition of

water, it will deposit in a difficultly soluble mass. The addition of cold water to the highly heated fused mass causes the sudden evolution of steam, with a sputtering which may be dangerous when manufacturing caramel; hence to insure perfect results, to the caramelized sugar at the proper stage should be added boiling water and not cold water.

In making sugar by the open kettle process there is obtained a large amount of brown mother liquor, and this contains a quantity of sugar in uncrystallized form, and the concentrated product is called *molasses*. In the modern manufacture of sugar the aim is to make as large an amount of sugar as possible, and therefore results in a corresponding loss in the yield of molasses. In the vacuum pan sugar-making the amount of molasses produced is very small, and even from this the sugar is extracted by treatment with strontium salts (which forms soluble strontium saccharate), from which the sugar can be eventually isolated in crystalline form.

Sugar, U. S. P., is a white, crystalline solid, of specific gravity 1.345, and soluble in 0.5 part of cold water, and sparingly soluble in alcohol. When treated with concentrated sulphuric acid the water is extracted, leaving carbon behind; in other words, sulphuric acid carbonizes sugar. Its saturated aqueous solution forms syrup, U. S. P. Sugar is chiefly used as a sweetener, possessing in this way decided nutritive properties.

SACCHARUM LACTIS. Sugar of Milk.



A peculiar crystalline sugar obtained from the whey of cows' milk by evaporation and purified by recrystallization.

White, hard, crystalline masses, or a white powder feeling gritty on the tongue, odorless, and having a faintly sweet taste. Permanent in the air. On incinerating a portion, the percentage of ash remaining should not exceed 0.25 per cent.

Soluble in 4.79 parts of water at 25° C. (77° F.) and in 1 part of boiling water; insoluble in absolute alcohol, ether, or chloroform.

An aqueous solution of Sugar of Milk is neutral to litmus-paper and is dextrogyrate.

On adding 5 Cc. of a hot, saturated aqueous solution of Sugar of Milk, an equal volume of sodium hydroxide T.S., and gently warming, the liquid will turn yellow and finally brownish-red. On the further addition of a few drops of cupric sulphate T.S., a brick-red precipitate will appear.

If about 1 Gm. of powdered Sugar of Milk be sprinkled upon about 5 Cc. of cold sulphuric acid (sp. gr. 1.84) contained in a flat-bottomed dish and covered with a watch-glass, the acid may acquire a greenish or reddish, but no brown or brownish-black color within half an hour (absence of *cane-sugar*).

An aqueous solution of Sugar of Milk (1 in 10) mixed with a few drops of hydrochloric acid should not respond to the Time-Limit Test for *heavy metals* (see p. 979).

If 1 Gm. of powdered Sugar of Milk be boiled for five minutes with 50 Cc. of distilled water and the solution cooled, no blue coloration should be produced upon the addition of one drop of iodine T.S. (absence of *starch*).

Tests for Identity.—Given above.

Impurities.—Cane-sugar, heavy metals, starch. Details above.

Milk-sugar is that form of biose chemically called lactose, and is a normal constituent of milk. Up to very recent years practically all milk-sugar was made in Switzerland, as, in the United States, the whey after cheese manufacture was used to feed cattle. At present, how-

ever, much sugar of milk is made in this country, most of it being produced in New York State.

Milk-sugar is made in Switzerland by simple concentration of whey in open kettles and then crystallizing on sticks. In this country the whey is evaporated in vacuo, and the moist granular mass dried in a centrifugal machine.

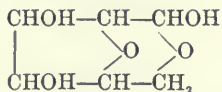
Milk-sugar appears either in hard white crystals or in fine white powder, with a taste not so sweet as that of cane-sugar. As mentioned above, it directly reduces Fehling's solution, while cane-sugar has no action on Fehling's solution until hydrolyzed. Another point of chemical distinction between the two products is shown in the test of the pharmacopœia, namely, that milk-sugar when sprinkled upon concentrated sulphuric acid should not blacken the same; if it did, the presence of cane-sugar is indicated. In carrying out this test great care should be exerted to use nothing but the pure powdered milk-sugar. If fragments of the stick or string on which it crystallized were present, such would produce a brown color with the acid.

Maltose is the sugar which is found in official *malt*. Malt is made from barley by placing same in a warm, moist, light place, and when the seeds begin to sprout, they are heated sufficiently to destroy the life of the young plants. The sprouting of a seed means the gradual development of embryo found therein, and during the first stages of its development the starch which is stored up in the seed serves for its food until there is developed a sufficient number of leaves to generate its own food from the carbon dioxide of the air. The starch which is found in the seed has to be converted into soluble forms before it can be assimilated by the embryo, and in the case of barley, it is converted into maltose by the action of the ferment *diastase* naturally residing in the seed. In manufacturing malt from barley the latter is allowed to grow until practically all the starch has been converted into maltose by the diastase. Maltose is of interest only as the sugar constituent of *extract of malt* (which see).

CARBOHYDRATES HAVING THE BASIC FORMULA



These furnish several well-known products, both cellulose and starch being examples. Both these bodies, however, are more complicated than the simple formula given above, and their composition is usually expressed by $(C_6H_{10}O_5)_x$. According to the investigation of Tollens, the x in the case of starch is 4, while it is assumed that the real formula of cellulose is $(C_6H_{10}O_5)_{12}$. Green and Perkin say that the graphic formula of cellulose is—



Cellulose is so called because it represents the coating of the cell. Cells consist primarily of a semiliquid portion called the protoplasm and nucleus surrounded by a cell-wall, and this cell-wall, when in a state of the greatest purity, consists of pure cellulose. A striking illustration of pure cellulose in the form of cell-wall is found in the official *cotton*, which consists of the hairs of the seed of *Gossypium*

herbaceum. In this case each hair consists of a single emptied cell, hence it is nothing but a cell-wall, and this, on chemical analysis, is shown to be pure cellulose.

Cotton consists of long filaments, insoluble in water, alcohol, and ether, but soluble in ammoniacal solution of copper oxide (not ammoniacal solution of copper sulphate, as mentioned in so many books). Cotton is used solely for mechanical purposes, and is of great value in pharmacy for such operations as filtering and straining, and in surgery as a dressing. The general use of cotton in the form of cloth is too well known to need remark.

GOSSYPIUM PURIFICATUM. Purified Cotton.

The hairs of the seed of *Gossypium herbaceum* Linné, or of other cultivated species of *Gossypium* (Fam. *Malvaceæ*), freed from adhering impurities and deprived of fatty matter.

White, soft, fine filaments, appearing under the microscope as hollow, flattened and twisted bands, spirally striate, and slightly thickened at the edges; inodorous and tasteless; insoluble in ordinary solvents, but soluble in an ammoniacal solution of cupric oxide.

When Purified Cotton, previously compressed in the hand, is thrown on the surface of cold water, it should readily absorb the latter and sink, and the water should not acquire either an acid or an alkaline reaction (evidence of *proper purification* and absence of *fatty matter*).

Purified Cotton should be perfectly free from all visible impurities, and on combustion should not leave more than 0.3 per cent. of ash.

Tests for Identity.—Given above.

Impurities.—Impure cotton, fatty matter. Details above.

Remarks.—This represents raw cotton freed from fat by boiling in a very dilute alkaline solution, and washing very thoroughly with water the first portion of which has been acidulated. It is commonly called *absorbent cotton*.

Raw cotton floats when placed in water, because each filament is coated with a layer of oil, rendering the cellulose impervious to water, which cannot, therefore, displace the air in the lumen (the cavity) of the cell. In this way each filament of raw cotton is buoyed up by the air within its lumen. Absorbent cotton is this raw cotton freed from its oily covering. Water is, therefore, able to penetrate the cellulose and thus displace the air in the lumen. The cellulose itself being heavier than water, the water-filled filament sinks; hence a pledget of absorbent cotton placed upon water immediately falls to the bottom of the vessel (see test given above).

Purified cotton is used as a surgical dressing and as a filtering medium.

Linen, the fibers of stem of the flax, and *silk*, the fibers of the cocoon of the silk-worm, are modifications of cellulose.

Paper originally represented a special form of shredded linen or cotton. The details of the manufacture of paper are beyond the scope of this book, but suffice it to say that the mass of shreds, when rolled out into paper, forms an absorbent sheet known as filter- or blotting-paper, according to its thickness. Writing paper represents this absorbent paper, which has been coated with a layer of some substance

which ink will not penetrate, such as a combination of glue with albumin, resin, or casein. Most paper now used in printing is, however, made from wood-fiber.

If cotton or linen paper is rapidly passed through 50 per cent. sulphuric acid, the outer layer of cells becomes converted into a modification (amyloid), forming the well-known product called *parchment paper*. If the paper is allowed to remain long in contact with sulphuric acid, it is converted into *dextrin*, which will dissolve in the acid. The same is true of absorbent cotton; it dissolves in concentrated sulphuric acid, but the solution in this case is different than is the solution of the cotton in ammoniacal copper oxide, because in the acid it dissolves not as cellulose, but as dextrin, whereas the copper solution is simply a solution of the cellulose from which the cotton can be redeemed by evaporation.

Cotton or cellulose, like glucose and the sugars, is an alcohol, as is proved by the fact that when treated with a mixture of nitric acid and sulphuric acid it is converted into the ester, nitrocellulose. Several forms of nitrocellulose are known, these being grouped into two classes—the first, true pyroxylin, and the colloxylin.

The first, which is the true *gun-cotton*, was discovered by Schoenbein in 1864, and has the formula $C_{12}H_{14}(ONO_2)_6O_4$, which, it will be noticed, contains six nitro-groups.

The second series, the colloxylin, are respectively:



containing five, four, and three nitro-groups. The true pyroxylin mentioned above is insoluble in a mixture of alcohol and ether, whereas the three colloxylin are soluble in a mixture of alcohol and ether.

PYROXYLINUM. Pyroxylin.

A product obtained by the action of nitric and sulphuric acids on cotton, and consisting chiefly of cellulose tetranitrate $[C_{12}H_{16}(ONO_2)_4O_6]$. It should be kept in cartons, protected from light.

A yellowish-white, matted mass of filaments, resembling raw cotton in appearance, harsh to the touch; exceedingly inflammable, burning, when unconfined, very rapidly with a luminous flame; less explosive than cellulose trinitrate.

Slowly but completely soluble in 25 parts of a mixture of 3 volumes of ether and 1 volume of alcohol. Soluble in acetone and in glacial acetic acid, and precipitated from these solutions on addition of water.

It should leave no weighable residue of mineral impurity when ignited.

When kept in well-closed bottles and exposed to the light, it is decomposed with the evolution of nitrous vapors, a carbonaceous mass being deposited.

Pyroxylin consists of a mixture of the three colloxylin (chiefly the tetranitrate) given above, and is made, as mentioned above, by immersing cotton in a mixture of nitric acid and sulphuric acid. An interesting fact in the manufacture worth pointing out is that the macerating cotton should be carefully covered, as it has been noticed that if a mixture is allowed to stand in a dish, particles of dust and the like are apt to start a new reaction in the mixture, with the ultimate formation of dextrin and complete solution of the pyroxylin.

The manufacture of pyroxylin is simple, but the freeing of the cotton from acid is tedious, and usually takes a large quantity of water for the purpose. If pyroxylin is not freed from acid, it will assume a brown tint and decompose. The same decomposition occurs if pyroxylin is kept in well-stoppered bottles, hence the pharmacopœia directs that it be kept in cartons.

The pyroxylin of the pharmacopœia has an appearance similar to that of cotton, but possesses a harsh feel, rather than the softness of the pure cotton. It should be completely soluble in a mixture of 1 part alcohol and 3 parts ether, and this solution, containing 3 per cent. of gun-cotton, is official under the name collodion (p. 209).

Gun-cotton is one of the most inflammable of substances, portions brought in contact with a flame instantly igniting with a very brilliant light, and leaving no trace of ash behind. Its great inflammability makes it a rather dangerous product to have around, and, therefore, it should be handled with the utmost caution and kept remote from heat or fire. If gun-cotton be heated in a confined space, the same decomposition occurs with explosive violence, and it is, therefore, used as an explosive.

Its action, however, is too uncertain to make it safe for this purpose; hence nitroglycerin is preferred for blasting rather than gun-cotton.

A mixture of pyroxylin with camphor, whitened with zinc oxide, and made pliable with castor oil, forms the well-known commodity *celluloid*. This possesses the same inflammability as pyroxylin, and, therefore, its use in various articles of apparel, such as combs, is not to be recommended.

Wood is cellulose which has become impregnated with other substances, the chief of which is presumably a nitroso-combination of an insoluble proteid. The botanic side of wood has been considered on p. 691, and it needs, therefore, to be discussed at this place only as a chemical. Of late years the demand for cheap paper has led to the use of wood for the manufacture of paper instead of linen and cotton. Such paper is by no means as durable as that from pure cellulose, and it is often convenient to be able to decide whether a given sample of paper contains wood-fiber. This is very beautifully determined by pouring on the suspected paper a solution of aniline acetate.

If the paper is pure cellulose, no change of color will be produced, but with a slight amount of wood-fiber the reagent will form a brilliant yellow. If paper is treated with a solution of phloroglucin, followed by a few drops of concentrated hydrochloric acid, a bright red stain results if wood-fiber is present.

Starches represent bodies having the formula $(C_6H_{10}O_5)_x$. They are formed in the leaves of plants from carbon dioxide and water, under the influence of chlorophyl, as already explained on p. 647, and represent the great plant reserve stuff.

During the time it is in leaf the plant manufactures starch, which is then stored up as nourishment for the young shoots of the following spring. While starch is formed in the leaves of plants, as these leaves are shed by the plant in the winter it is necessary that starch be stored in some other organ during that period; hence the starch formed in the leaves is transported to the organs in which it will be reserved (very

frequently the roots or rhizome), where it is changed back into starch. Likewise in the spring, the stored matter is converted once more into sugar, which passes in solution up to the growing part of the plant. This solution is known as "sap," and commercial advantage is taken of the sugar contents of sap in the preparation of *maple-sugar*.

Starch is found in various parts of the plant, in particles called starch-granules. The appearance of these granules is characteristic for the different varieties of starch, and some are shown in Fig. 225.

The starches are usually soluble in cold water, and on being treated with hot water swell into a paste, which is more or less soluble in water. The test for starch is that the addition of a drop of iodine to starch paste turns it blue, and if too much iodine is added, it becomes black.

As mentioned above, starch is converted by the ferment diastase into maltose (p. 699), while the same starch treated with sulphuric acid is converted into glucose.



Fig. 225.—Microscopic appearance of different starches (uniform magnitude) (Noël): 1, Arrowroot; 2, raw tapioca; 3, tapioca.

The most abundant starch is that from potato, from which it is extracted by a very simple process, grated potato being poured into troughs over which water is allowed to run, the water dissolving out the albuminates and other soluble principles, while the starch settles in the bottom of the trough in which it is collected. This potato starch is the well-known starch used in laundries, the starch for food or medicine being obtained either from corn or wheat.

AMYLUM. Starch. Corn Starch.

The starch grains obtained from the fruit of *Zea Mays* Linné (Fam. *Gramineæ*).

In fine powder or irregular, angular white masses, consisting of somewhat spherical, but usually polygonal grains, about 0.010 to 0.025 Mm. in diameter, with a lenticular, circular, or triangular central fissure; inodorous and tasteless; insoluble in cold water or alcohol; forming a whitish jelly when boiled with water, which when cool gives a deep blue color with iodine T.S.; triturated with cold water, showing neither acid nor alkaline reaction with litmus-paper; when completely incinerated, leaving not more than 1 per cent. of ash.

When freed from water by careful drying in a current of warm air, Starch should show not less than 95 per cent. of hydrolyzable carbohydrate.

The official starch is that from corn. As seen in the picture, starch-granules from the same source are similarly marked, having more or less well-defined concentric rings radiating from a central spot called the hilum.

Starch Modifications.—*Lichenin* is a variety of starch found in Irish moss. It does not turn iodine blue.

Inulin is a starch modification found in roots of the natural order Compositæ. It differs from starch in being soluble in water, from which it separates in crystalline form and turns iodine yellow instead of blue.

Glycogen is a starch modification which is of interest only because of its animal origin, being found in the liver.

GUMMY SUBSTANCES

Gums have a composition similar to starch. They differ from starch in being either soluble in cold water or swelling to a jelly therein, and in being precipitated from aqueous solution by alcohol. On oxidation with nitric acid they yield mucic acid, while starch on oxidation with nitric acid yields dextrin.

Among the gummy principles of pharmaceutic importance are the following:

Dextrin, made by oxidation of starch with nitric acid, this being the commercial method of manufacture.

It is also produced by simply heating starch, and it is of passing interest to note that the polish we see on the outer surface of a loaf of bread is due to the dextrinization of the starch of the flour, and to the same cause is due the high polish acquired by ironing well-starched linen.

Dextrin is a whitish-yellow body, dissolving in water to form mucilage, for which purpose it is chiefly employed. On account of the high price of gum arabic, dextrin has been suggested as an emulsifier in making cod-liver oil, and, in fact, a recipe for making an emulsion with dextrin can be found in the National Formulary. Such emulsions have never yielded satisfactory results in the writer's experience.

Arabic Acid.—This is the active principle of acacia, where it is found as the calcium salt (*arabin*). On hydrolysis it yields glucose and arabinose, the latter a carbohydrate having the formula $C_5H_{10}O_5$.

Bassorin is the chief constituent of gum tragacanth. It differs from dextrin in the fact that it does not dissolve in cold water, but simply swells to form mucilage.

Cerasin, or fruit-gum, is the well-known exudation from peach and plum trees. It is insoluble in water, but dissolves in alkali.

Pectins are acid combinations of carbohydrates. They are found in various vegetable juices, and under the influence of a ferment, *pectase*, they coagulate. This explains the manufacture of jellies from fruit-juices, and also explains why preparations of some drugs, notably senega, gelatinize. Bertrand and Mallèvre found that calcium, or one of the other alkaline earths, is necessary to produce gelatinization, proving that the coagulated mass is the calcium salt of pectic acid, and not pectic acid itself.

SACCHARINE DRUGS

MANNA. *Manna*.

The concrete saccharine exudation of *Fraxinus Ornus* Linné (Fam. *Oleaceæ*).

Constituents.—Mannite, sugar, dextrin. *Fraxin*, $C_{16}H_{18}O_{10}$.

Remarks.—Manna exudes from the trunk of the manna ash during the heat of the day, solidifying in the chill evening air. The pharma-

copœia directs the use of what is commercially called "large-flake" or "small-flake" manna, "sorts" manna being rejected. A favorite method of administration is as "senna, manna, and salts" (compound infusion of senna, p. 215).

Medical Properties.—Laxative.

Dose.—16 Gm. (240 grains).

MALTUM. Malt.

The grain of barley, *Hordeum distichon* Linné (Fam. *Gramineæ*), partially germinated artificially, and then dried.

Constituents.—Maltose and diastase.

Remarks.—Malt is barley-seed permitted to begin to sprout when the insoluble starch is converted by the ferment diastase into soluble sugar (maltose), that it can be of service in feeding the young plant. So soon as this conversion is accomplished the plant life is destroyed by heating to 70° C. Malt is official merely because used for making the official extract of malt (p. 285).

MEL. Honey.

A saccharine secretion deposited in the honeycomb by the bee, *Apis mellifera* Linné.

A syrupy liquid of a light yellowish to yellowish-brown color, translucent when fresh, but gradually becoming opaque and crystalline, having a characteristic, aromatic odor, and a sweet, faintly acid taste.

When recent Honey is diluted with twice its weight of water, the resulting liquid should be almost clear, not stringy, and should have a specific gravity not lower than 1.099 (corresponding to a specific gravity of 1.370 for the original Honey).

When Honey is incinerated in small portions at a time in a platinum crucible, it should not leave more than 0.3 per cent. of ash.

Honey has a faintly acid reaction upon litmus-paper and is lævogyrate.

If 2 Cc. of a filtered solution of the Honey (1 in 4) be placed in a test-tube 1 Cm. in diameter, and 1 Cc. of absolute alcohol be allowed to flow down the walls of the tube held in an inclined position, so as to form an overlying layer, this should remain clear, and the line of contact should not show more than barely noticeable opalescence, which soon disappears; a permanent milky zone should not be produced (absence of starch sugar).

If 2 Cc. of pure concentrated sulphuric acid be placed in a test-tube of 1 Cm. diameter, and 0.5 Cc. of a solution of Honey (1 in 4) be allowed to flow upon it so as to form a distinct upper layer, no colored line of contact should show at once, and at the end of one hour the zone of contact should be at most yellowish or clear brown; a brownish color becoming nearly black at the end of half an hour should not develop (absence of cane-sugar).

On boiling 1 part of Honey with 5 parts of water, the resulting solution, when cold, should not be rendered blue or green on the addition of iodine T.S. (absence of starch).

Tests for Identity.—Given above.

Impurities.—Starch-sugar, cane-sugar, and starch. Details above. Chlorides and sulphates. Details in Part V.

Constituents.—Dextrose, levulose, traces of cane-sugar, and dextrin, particles of wax.

Remarks.—Honey usually represents the nectar extracted from flowers by the bee and stored in the honeycomb for food purposes.

That the bee is able to produce a chemical change in the nectar in transferring it from the flower to the comb is shown by the ability of

captive bees to convert dextrogyrate sugar to levogyrate honey. The tests for honey given above are important, as commercial honey is notoriously adulterated with artificial glucose (starch sugar). The barium chloride test has a bearing on this point, since most commercial glucose contains traces of sulphates from the sulphuric acid used in producing it from starch.

For *clarified honey*, U. S. P., see p. 203.

Medical Properties.—Practically none, save as a pharmaceutic vehicle and as an excipient.

Dose.—4 Cc. (1 fluidrachm).

PRUNUM. Prune.

The partly dried ripe fruit of *Prunus domestica* Linné (Fam. *Rosaceæ*).

Constituents.—Sugar, malic acid, pectin.

Medical Properties.—Laxative, nutritive. Official only because an ingredient of confection of senna.

CASSIA FISTULA. Cassia Fistula.

The dried fruit of *Cassia Fistula* Linné (Fam. *Leguminosæ*).

Constituents.—The pulp consists of sugar, gum, pectin

Remarks.—This long pod, called “purging cassia,” is official because it is an ingredient of confection of senna.

Medical Properties.—Laxative.

Dose.—4 Gm. (60 grains).

FICUS. Fig.

The partially dried fruit of *Ficus Carica* Linné (Fam. *Moraceæ*).

Constituents.—Sugar, gum.

Remarks.—Of the fig, the only true fruit part is the tiny seed “seeds” (or achenes). The so-called fruit is the basis (receptacle), upon the hollow interior surface of which rest the separate minute flowers and the fruit produced therefrom. Such fruit is called a syconium.

Medical Properties.—Demulcent, laxative, and nutritive. Official because a constituent of confection of senna.

TRITICUM. Triticum.

The dried rhizome of *Agropyron repens* (Linné) Beauvois (Fam. *Gramineæ*), gathered in the spring.

Constituents.—Levulose, tritacin, malates, mucilage.

Remarks.—This drug is known as *couch grass* and *quick grass*. It has already been discussed as yielding a fluidextract made with aqueous menstruum (p. 252).

Medical Properties.—Diuretic.

Dose.—8 Gm. (120 grains).

MUCILAGINOUS DRUGS

A mucilaginous drug is one containing one of the gums, discussed from the chemical standpoint on p. 704. To what is there stated need only be added the warning that many substances commonly called "gums" are not true gums, in the modern pharmaceutic sense. Thus "gum elastic" (or rubber) and "chicle gum" (the basis of modern chewing-gum) are dried milk-juices; "gum asafetida" (p. 856) and "gum myrrh" (p. 857) are gum resins; "gum camphor" (p. 810) is the solid part of a volatile oil; "gum turpentine" (p. 845) is an oleo-resin; "gum tolu" (p. 850) is a balsam; "gum mastic" (p. 853) and "gum benzoin" (p. 855) are resins, and so on.

What these substances—milk-juices, gum resins, oleoresins, balsams, etc.—really are will be explained in the appropriate place, and here let it be said that a true gum is a carbohydrate derivative, more or less soluble in water (forming an adhesive solution or paste) and insoluble in alcohol.

ALTHÆA. *Althæa*.

The dried root of *Althæa officinalis* Linné (Fam. *Malvaceæ*), collected from plants of the second year's growth, and deprived of the periderm.

Constituents.—Mucilage, asparagin (p. 684), starch, sugar, pectin.

Remarks.—The "periderm" referred to in the official definition is the outer coating—the so-called "peel" or "bark." The drug is commonly called "marshmallow root," and it might be said, in passing, that the confection, "marshmallow drops," originally contained this drug. Now it is innocent of such admixture, being made from sugar and gum dusted with cornstarch. Powdered althæa is a valuable absorbent medium for making pills from liquids.

Medical Properties.—Demulcent, usually in the form of syrup (syrupus althææ, U. S. P. 1890, p. 193).

ULMUS. *Elm*.

The dried bark of *Ulmus fulva* Michaux (Fam. *Ulmaceæ*), deprived of its periderm.

Constituents.—Mucilage, trace of tannin, and only a very little starch.

Remarks.—Ulmus or *slippery-elm bark* is the inner bark of the special kind of elm tree, the periderm or corky bark having been removed.

Medical Properties.—Demulcent and emollient. The mucilage (mucilago ulmi, p. 189) is used as a basis of domestic cough remedies, while the ground bark is used as a soothing poultice.

SASSAFRAS MEDULLA. *Sassafras Pith*.

The dried pith of *Sassafras variifolium* (Salisbury) O. Kuntze (Syn. *Sassafras Sassafras* (Linné) Karsten) (Fam. *Lauraceæ*).

Constituents.—Mucilage which is not precipitated from aqueous solution by alcohol.

Medical Properties.—Demulcent. The infusion (infusum sassafras medullæ, U. S. P. 1890) is used as a soothing basis for eye lotions.

ACACIA. Acacia.

A gummy exudation from *Acacia Senegal* Willdenow, and other species of *Acacia* (Fam. *Leguminosæ*).

Constituents.—Arabin, a levogyre gum, chiefly calcium salt of arabic acid, which hydrolyzes to arabinose, $C_5H_{10}O_5$; also a dextrogyre gum which hydrolyzes to glucose.

Remarks.—Acacia is precipitated from solution by lead subacetate, ferric chloride, and saturated solution of borax; hence these substances should not be prescribed with mucilage of acacia; alcohol also precipitated the mucilage.

Medical Properties.—Demulcent. It is the most important pharmaceutical adhesive agent, and is employed for this purpose in emulsions. Official preparations: Syrup of acacia (p. 192) and mucilage of acacia (p. 188).

TRAGACANTHA. Tragacanth.

A gummy exudation from *Astragalus gummifer* Labillardière, or from other species of *Astragalus* (Fam. *Leguminosæ*).

Constituents.—Water-soluble gum, similar to arabin. Bassorin, $C_{12}H_{20}O_{12}$, which does not dissolve in water, but simply swells therein, making a jelly.

Remarks.—Tragacanth differs from acacia in swelling to a jelly, when mixed with water, instead of dissolving therein, as does acacia. The official mucilage (*mucilago tragacanthæ*, p. 189) is largely used as a basis for jellies for chapped hands. It is also an excellent pill excipient.

Medical Properties.—Demulcent.

CHONDRUS. Chondrus.

The dried plant of *Chondrus crispus* (Linné) Lyngbye (Fam. *Gigartinaceæ*).

Constituents.—Pectin (called *carragheen* by Pareira), mucus, and traces of iodine and bromine.

Medical Properties.—Chondrus (*Irish moss* or *carragheen*) is used as a demulcent and nutritive, being always administered in the form of a jelly made by boiling one part of drug with thirty parts of water and letting strained solution cool. The decoction, carefully evaporated, and the syrupy residue poured on glass plates, yields scaled *Irish moss gelatin*, recommended by Emlen Painter as an emulsifying base.

Dose.—16 Gm. (4 drachms).

Cetraria, the lichen, *Cetraria islandica*, was official in former pharmacopœias, but was dropped in the revision of 1890. It contains two mucilaginous principles, *lichinin*, $C_{12}H_{20}O_{10}$, and *lichnoid*, and a bitter principle, *cetraric acid*, since discovered to be a mixture. It was used as a nutritive in sick diet in the form of jelly (*decoctum cetrariæ*, U. S. P. 1890). For this purpose, however, the bitter cetraric acid had to be removed before jelly was made; hence the directions in making the decoction to throw away the first water in which the drug was washed (p. 216).

CHAPTER XL

GLUCOSIDES AND GLUCOSIDAL DRUGS

THE large and important class of plant principles called glucosides are ethers of glucose with complex bodies, the usual definition of glucosides being *bodies which, when treated with a diluted acid or a ferment, split into glucose and some other body*.

This "other body" is usually of complex character, the composition of which is little known. In most cases the glucoside derivatives are compounds of benzene, and as the knowledge of these derivatives is extended and they are placed in their proper position in systematic chemistry, the glucoside in question can be more properly studied under such heading. Already we are confronted with this condition in the glucosidal drugs bitter almond and black mustard. In each case the product of hydrolysis is of more pharmaceutic importance than is the glucoside itself, hence black mustard is studied among the thiocyanates (p. 746), while the bitter almond is considered along with the benzaldehydes (p. 778).

Even the word "glucose," used in the above definition, does not indicate the definite chemical we know by that name. As already learned, there are a large number of bodies having the formula $C_6H_{12}O_6$, and "glucose" in the above definition means any one of these. More than that, some glucosides yield carbohydrates other than hexoses; thus, digitalin is said to yield digitalose, $C_7H_{14}O_5$.

The word "*hydrolysis*" used above expresses the process of decomposing chemical substances by introducing the element of water, this being the function of the diluted acid or the ferment in the decomposition of glucosides, as expressed in the following equations, illustrative of a few examples of such decomposition of glucosides:

Amgaldin plus Water equal Glucose plus Hydrocyanic acid plus Benzaldehyde.

$$C_{20}H_{27}NO_{11} + 2H_2O = 2C_6H_{12}O_6 + HCN + C_6H_5CHO$$

Arbutin plus Water equal Glucose plus Hydroquinone plus Methyl hydroquinone.

$$C_{25}H_{34}O_{14} + 2H_2O = 2C_6H_{12}O_6 + C_6H_4(OH)_2 + CH_3C_6H_3(OH)_2$$

Convolvulin plus Water equal Glucose plus Methylethyl plus Penta-decylic acid.

$$C_{32}H_{62}O_{16} + H_2O = 2C_6H_{12}O_6 + C_5H_{10}O_2 + C_{15}H_{30}O_3$$

Digitalin equals Digitaligenin plus Glucose plus Digitalose.

$$C_{35}H_{50}O_{14} = C_{22}H_{30}O_3 + C_6H_{12}O_6 + C_7H_{14}O_5$$

Digitonin plus Water equal Digitogenin plus Galactose plus Dextrose.

$$C_{27}H_{44}O_{13} + 2H_2O = C_{15}H_{24}O_3 + C_6H_{12}O_6 + C_6H_{12}O_6$$

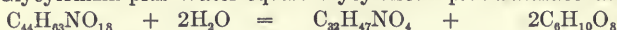
Digitoxin plus Water equal Digitoxigenin plus Digitoxose.

$$C_{34}H_{54}O_{11} + H_2O = C_{22}H_{32}O_4 + 2C_6H_{12}O_4$$

Gentiopiecin plus Water equal Gentiogenin plus Glucose.

$$C_{16}H_{26}O_9 + H_2O = C_{16}H_{16}O_4 + C_6H_{12}O_6$$

Glycyrrhizin plus Water equals Glycyrrhetin plus Parasaccharic Acid.



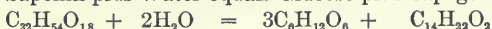
Myronic Acid gives Glucose plus Potassium Bisulphate plus Oil Mustard.



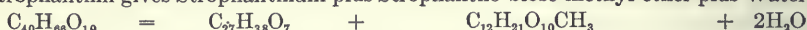
Salicin plus Water equals Glucose plus Saligenin.



Saponin plus Water equals Glucose plus Sapogenin.



Strophanthin gives Strophanthidin plus Strophantho-biose-methyl-ether plus Water.



The following glucosides have been studied, but not sufficiently to furnish complete equation:

Chiratin hydrolyzes to ophelic acid and chiratogenin.

Colocynthin hydrolyzes to colocynthein and sugar.

Convallarin hydrolyzes to convallaretin.

Convallamarin hydrolyzes to convallamaretin.

Eriocolin hydrolyzes to ericinol.

Parillin hydrolyzes to parigenin.

Scammonin hydrolyzes to scammonol, valerianic acid, and grape sugar.

SAPONINS

Under the head saponin we include a large number of glucosides frothing like soap when shaken with water, usually toxic, and while closely similar, are not identical in chemical composition, nor is the hydrolysis product, sapogenin, identical in each case. The following list gives the composition of the known saponins as reported by Kobert:

Toxic saponins:

Quillajic acid.....	$\text{C}_{18}\text{H}_{25}\text{O}_{10}$, from quillaja.
Quillaja sapotoxin.....	$\text{C}_{17}\text{H}_{20}\text{O}_{10}$, from quillaja.
Parillin.....	$\text{C}_{26}\text{H}_{44}\text{O}_{10}$, from sarsaparilla.
Sarsasaponin.....	$\text{C}_{22}\text{H}_{36}\text{O}_{10}$, from sarsaparilla.
Smilasaponin.....	$\text{C}_{20}\text{H}_{32}\text{O}_{10}$, from sarsaparilla.
Senegin.....	$\text{C}_{17}\text{H}_{26}\text{O}_{10}$, from senega.
Polygalic acid.....	$\text{C}_{18}\text{H}_{28}\text{O}_{10}$, from senega.
Melanthinic acid.....	$\text{C}_{29}\text{H}_{50}\text{O}_{10}$, from veratrum.

Non-toxic saponins:

Chamælrin.....	$\text{C}_{36}\text{H}_{63}\text{O}_{18}$ from Helonias dioica.
Two saponins from guaiac	$\text{C}_{21}\text{H}_{34}\text{O}_{10}$ $\text{C}_{22}\text{H}_{36}\text{O}_{10}$

It will be noted that all the formulas just given conform to either one of the two general formulas. Those containing 18 atoms of oxygen agree with the formula proposed by Flückiger, $\text{C}_n\text{H}_{2n-10}\text{O}_{18}$, while those containing 10 atoms of oxygen agree with the general formula suggested by Kobert, $\text{C}_n\text{H}_{2n-8}\text{O}_{10}$.

In passing, let it be said that the formula for saponin, found in the hydrolysis equation given above, which does not agree with any formula in list of saponins just given, is one that conforms to the Flückiger general formula.

GLUCOSIDAL DRUGS

As mentioned above, glucosides owe their characteristics primarily to those products of hydrolysis other than glucose, and as these products possess widely varying properties, the grouping of glucosidal drugs brings together agents showing marked difference in medical

action. In an attempt to bring a semblance of order into this therapeutic confusion the drugs considered in this chapter will be grouped as follows:

Drug containing sweet glucosides.....	Glycyrrhiza.
Drugs containing bitter glucosides.....	Gentian, chirata, scutellaria, saffron (U. S. P. 1890), and salicin.
Drugs containing glucosides acting on heart....	Convallaria, strophanthus, squill, and digitalis.
Drugs containing purgative glucosides.....	Lappa, jalap, scammony, colocynth.
Drugs containing astringent glucosides.....	Uva ursi, chimaphila.
Drugs containing saponin.....	Quillaja, sarsaparilla, senega.

GLYCYRRHIZA. Glycyrrhiza. Licorice Root.

The dried rhizome and root of *Glycyrrhiza glabra* Linné (Spanish Licorice), or of *Glycyrrhiza glandulifera* Waldstein and Kitaibel (Russian Licorice) (Fam. *Leguminosæ*).

Constituents.—*Glycyrrhizin* or *glycyrrhizic acid*, the ammonium salt being the combination found in the drug; glycyrrhamarin; starch.

Remarks.—The pharmacopœia permits the use of both Spanish and Russian licorice, the latter being larger than the Spanish, and comes into commerce peeled. It is sweeter than the Spanish, hence preferable.

Medical Properties.—Demulcent and expectorant. Used largely to disguise the taste of quinine and other bitter substances, for which purpose *ammoniated glycyrrhizin* or the *fluidextract* (p. 251) is used.

The other official preparations of glycyrrhiza are the two extracts (p. 284), brown mixture (p. 265), and compound licorice powder (p. 303).

Dose.—2 Gm. (30 grains).

GLYCYRRHIZINUM AMMONIATUM. Ammoniated Glycyrrhizin.

Recipe.—Glycyrrhiza, in No. 20 powder, five hundred grammes..... 500 Gm.

Water,
Ammonia Water,
Sulphuric Acid, each, a sufficient quantity.

Mix four hundred and seventy-five cubic centimeters of Water with twenty-five cubic centimeters of Ammonia Water, and, having moistened the powder with the mixture, macerate for twenty-four hours. Then pack it moderately in a conical glass percolator, and gradually pour Water upon it until five hundred cubic centimeters of percolate are obtained. Add Sulphuric Acid slowly to the percolate, with constant stirring, so long as a precipitate is produced. Collect this on a strainer, wash it with cold Water until the washings no longer have an acid reaction, redissolve it in Water with the aid of Ammonia Water, filter, if necessary, and again add Sulphuric Acid so long as a precipitate is produced. Collect this, wash it, dissolve it in a sufficient quantity of Ammonia Water previously diluted with an equal volume of Water, and spread the clear solution upon plates of glass, so that, when dry, the product may be obtained in scales.

Dark brown or brownish-red scales, without odor, and having a very sweet taste.

Readily soluble in water and alcohol.

The aqueous solution, when heated with potassium hydroxide T.S., evolves ammonia.

If the aqueous solution be supersaturated with an acid, there will be produced a precipitate (glycyrrhizin) which, when dissolved in hot water, forms a jelly on cool-

ing. This substance, after being washed with diluted alcohol and dried, appears as an amorphous, yellow powder, having a strong, bitter-sweet taste, and an acid reaction.

Upon incineration, Ammoniated Glycyrrhizin should not leave more than a trace of ash.

Remarks.—This substance, representing the glycyrrhizin in more or less impure form, is made by precipitation from an ammoniacal aqueous percolate of glycyrrhiza by means of sulphuric acid. The crude precipitated glycyrrhiza is dissolved in ammonia water and water, is reprecipitated with acid, again dissolved in ammonia water and water, and then "scaled" (p. 657).

Medical Properties.—This product is used entirely for masking taste of bitter substances.

Dose.—250 milligrammes (4 grains).

GENTIANA. Gentian.

The dried rhizome and roots of *Gentiana lutea* Linné (Fam. *Gentianaceæ*).

Constituents.—*Gentiopicroin*, a bitter glucoside; *gentianin*, a bitter principle; *gentisin*, when fused with potassa, yields *gentisic acid*, a body that turns deep blue with ferric chloride, etc.

Gentian contains no tannin, the darkening of its preparations by iron salts being due to gentisin and gentisic acid.

Remarks.—Gentian preparations yield unsightly mixtures when combined with iron unless the gentisin is removed by treatment of the preparation with ferric hydroxide.

This process is used in making Elixir of Gentian, N. F. A still more popular preparation of gentian is the glycerinated Elixir of Gentian, N. F., which is similar to the very popular but wrongly named "glycerin tonic."

Medical Properties.—Simple bitter, stimulates digestion and improves the appetite.

Administered as *fluidextract* (p. 247) and as *compound tincture of gentian* (p. 227). The official *extract* (p. 283) is a useful excipient for tonic pills, but should not be used for this purpose by a pharmacist unless so prescribed by the physician.

Dose.—1 Gm. (15 grains).

CHIRATA. Chirata.

The dried plant of *Swertia Chirayita* (Roxburgh) Hamilton (Fam. *Gentianaceæ*).

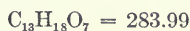
Constituents.—*Ophelic acid* and *chiratin*, the latter a glucoside.

Remarks.—Chirata is an East Indian herb of intensely bitter taste, little used in this country, but quite popular in England.

Medical Properties.—A simple bitter.

Dose.—1 Gm. (15 grains).

SALICINUM. Salicin.



A glucoside obtained from several species of *Salix* and *Populus* (Fam. *Salicaceæ*). Salicin should be kept in well-stoppered bottles.

Colorless, silky, shining, crystalline needles, rhombic prisms, or a white crystalline powder; odorless and having a very bitter taste.

Soluble in 21 parts of water and in 71 parts of alcohol at 25° C. (77° F.), in 3.3 parts of water at 80° C. (176° F.), and in 22 parts of alcohol at 60° C. (140° F.); insoluble in ether and chloroform.

It melts at 201.4° C. (394.5° F.). Upon ignition it is consumed, leaving no residue.

Its aqueous solution is laevogyrate and neutral to litmus-paper.

On heating a small portion of Salicin in a test-tube until it turns brown, then adding a few Cc. of water, and afterward a drop of ferric chloride T.S., a violet color will be produced.

Sulphuric acid produces a red color, which disappears upon the addition of water.

Upon gently heating 0.1 Gm. of Salicin with 0.2 Gm. of potassium dichromate and 2 Cc. of diluted sulphuric acid, the odor of salicylic aldehyde will be developed.

Sulphuric acid containing a trace of molybdic acid produces with Salicin a violet color, changing to a deep brownish-red. Sulphuric acid containing a trace of potassium iodate produces a dark red color, changing to deep purple. Sulphuric acid containing about one-fifth of its volume of solution of formaldehyde produces a deep purplish-red color.

If to a small quantity of Salicin a few drops of nitric acid be added, the liquid evaporated to dryness, and the resulting yellowish residue treated with ammonia water and heated upon a water-bath with a fragment of potassium cyanide, a blood-red color will be developed.

The aqueous solution of Salicin is not precipitated by tannic or picric acid T.S. nor by mercuric potassium iodide T.S. (absence of, and difference from, *alkaloids*).

Tests for Identity.—Given above.

Color Reactions.—Given above.

Impurities.—*Alkaloids.* Details above.

Remarks.—Salicin is prepared by making an aqueous infusion of willow bark, precipitating the gum and starchy matter from the infusion by treatment of lead subacetate, removing the excess of lead acetate by treatment with calcium carbonate. The filtrate is then concentrated in order to permit the crystals of salicin to separate out. These are purified by recrystallization.

Remarks.—As mentioned at the beginning of this chapter, salicin hydrolyzes to saligenin. This later oxidizes to salicylic aldehyde and salicylic acid (p. 782), and explains dichromate—sulphuric acid reaction given above.

Medical Properties.—Febrifuge and antirheumatic.

Dose.—1 Gm. (15 grains).

SCUTELLARIA. *Scutellaria*. Skull Cap.

The dried plant of *Scutellaria lateriflora* Linné (Fam. *Labiata*).

Constituents.—A glucoside, *scutellarin*; a trace of volatile oil.

Medical Properties.—*Stomachic, tonic*; popularly supposed of value as a nervine.

Dose.—1 Gm. (15 grains).

Saffron (U. S. P. 1890).—This drug is interesting as one of the most expensive of vegetable drugs, its great cost being due to the labor of collection. The definition of the drug is "the stigmas of *Crocus sativus*," by which is meant the upper portion of the female organ of the flower.

On taking an Easter lily or Bermuda lily and slicing lengthwise the white, funnel-shaped petal group (called the perianth) and removing it, all the interior organs are removed save the large central club-shaped column. This is the pistil or female organ, and this consists of three parts: First, the ovary at the base, which, on

being sliced, shows the ovules which develop into seeds; second, the style or slender tube, terminating in, third, a disc-like triple divided stigma designed to hold the pollen by which the ovary is fertilized.

In saffron, the stigma, instead of being a flat disc like that of the lily, consists of three delicate threads, and these constitute the official drug, to obtain a pound of which necessitates the picking of stigmas of 30,000 to 40,000 flowers, and that by hand. Hence, it is easy to understand why saffron is so expensive and why so largely adulterated, the chief adulterant being the shredded petals of other flowers. The chief use of saffron is in domestic practice—for measles—and in cooking.

CONVALLARIA. *Convallaria*. Lily of the Valley.

The dried rhizome and roots of *Convallaria majalis* Linné (Fam. *Liliaceæ*).

Constituents.—*Convallarin*, a glucoside; *convallamarin*, a saponin.

Medical Properties.—Slows and strengthens heart-beat, as does digitalis. Administered as fluidextract (p. 246).

Dose.—500 milligrammes (7½ grains).

STROPHANTHUS. *Strophanthus*.

The ripe seed of *Strophanthus Kombe* Oliver (Fam. *Apocynaceæ*), deprived of its long awn.

Constituents.—*Strophanthin*, a glucoside; *kombic acid*, a fixed oil, etc.

Remarks.—The “awn” referred to in the official definition is a feather-like appendage to the seed, intended to aid in transportation of the seed by the wind.

The fixed oil is a source of annoyance to the pharmacist, as a tincture made with strong alcohol becomes milky when mixed with water. In fact, when first introduced it was advised to remove the oil by percolation with ether before preparing the tincture. It has since been found that the active principles are dissolved by a diluted alcohol (650 parts alcohol to 350 parts water), which at the same time prevents solution of the oil. This is the menstruum used in preparing the official tincture (p. 236).

Medical Properties.—Acts on the heart as does digitalis.

Dose.—65 milligrammes (1 grain).

STROPHANTHINUM. *Strophanthin*.

A glucoside, or mixture of glucosides, obtained from *Strophanthus*. It should be kept in well-stoppered, amber-colored vials.

A white or faintly yellowish crystalline powder, containing varying amounts of water of crystallization, which it does not lose entirely without decomposition. Taste intensely bitter; *great caution should be used in tasting it*. Permanent in the air.

It is very soluble in water and in diluted alcohol, less soluble in absolute alcohol, but nearly insoluble in ether, chloroform, and benzene.

When heated, it begins to fuse at 170° C. (338° F.), and is not completely melted until the temperature of 190° C. (374° F.) is reached.

Its solutions are dextrogyrate and neutral to litmus-paper.

Sulphuric acid produces with *Strophanthin* an emerald-green color, changing to brown.

If to an aqueous solution of *Strophanthin* a trace of ferric chloride T.S. and a few Cc. of sulphuric acid be added, a red-brown precipitate will be produced, turning dark green after one or two hours.

Strophanthin should not reduce alkaline cupric tartrate V.S.; if its solution be heated to 70° C. (158° F.) with a small amount of diluted hydrochloric acid (1 in 20), it will be decomposed into strophanthidin, which precipitates, and a sugar, which will remain in solution, and which will reduce alkaline cupric tartrate V.S.

Tests for Identity.—Given above.

Color Reactions.—Given above.

Manufacture.—Strophanthin is made by extracting the fat-free pulverized seeds with 70 per cent. alcohol, removing the alcohol from the tincture by distillation, dissolving the residue in water, and precipitating the filtrate with tannic acid. This precipitate is mixed with litharge, dried, exhausted with alcohol, and the pure strophanthin is precipitated from this alcoholic solution by the addition of ether.

It will be seen from the above definition that it is a question whether strophanthin is a definite body or a mixture. It is used like strophanthus, but in very minute doses.

Dose.— $\frac{3}{10}$ milligramme ($\frac{1}{200}$ grain).

SCILLA. Squill.

The bulb of *Urginea maritima* (Linné) Baker (Fam. *Liliaceæ*), deprived of its dry, membranaceous outer scales, cut into thin slices and carefully dried, the central portions being rejected.

Constituents.—*Scillipicrin*, *scillitoxin*, *scillin*. Of these principles but little is known. Squill contains calcium oxalate in peculiar-shaped crystals called *raphides*, and also mucilage.

Remarks.—While squill is used as a domestic remedy, it must be borne in mind that its active principles, particularly scillipicrin, have action on the heart similar to digitalis. Hence squill preparations should be used with caution. By reason of the irritating action of its calcium oxalate crystals, ground squill has been recommended as a rat poison.

Medical Properties.—Nauseant, expectorant, and diuretic.

Official Preparations.—The *vinegar* (p. 263), the *syrup* (made from the vinegar) (p. 200), the *fluidextract* (p. 252), and the *compound syrup*, which is made from the fluidextract (p. 200). *Tincture of squill*, U. S. P. (p. 236) is made with an alcoholic menstruum.

Dose.—125 milligrammes (2 grains).

DIGITALIS. Digitalis.

The dried leaves of *Digitalis purpurea* Linné (Fam. *Scrophulariaceæ*), collected from plants of the second year's growth, at the commencement of flowering.

Constituents.—*Digitonin* (a saponin), *digitalin* (a glucoside), *digitalein* (mixture of several bodies, chiefly digitonin), *digitoxin*, *digitophyllin*, *digitin*, *digitoflavin*, 5 per cent. fat, and a trace of substance darkening with ferric chloride. Of these, the first four are the only ones of therapeutic importance, and, in truth, the real activity of digitalis seems included in the two principles—digitalin, the cardiac stimulant, and digitonin, depressant and diuretic.

Remarks.—The phrase "plants of the second year's growth," found in the official definition, indicates that the plant, *Digitalis purpurea*, is a biennial, lives two years, dying down to the ground in the fall of the first year, and in the following spring sending up a new batch of leaves and a flowering stalk from the roots which have lain dormant underground during the winter.

It is these second-year leaves that are active, and these only should be used in medicine.

It will be seen from the mention of the constituents given above that the chemistry of digitalis is very complex, and is not yet fully cleared up. The confusion is particularly noted in the various digitalins found in the market—the so-called “French digitalin,” of Nativelle; that of Homolle; and the commercial “German digitalin,” being totally different bodies. The last consists practically of digitonin, and is of but little activity (dose, $\frac{1}{10}$ to $\frac{1}{2}$ grain); while Homolle’s is quite potent (dose, $\frac{1}{80}$ to $\frac{1}{40}$ grain). Digitoxin is now considered a more important heart tonic than the several digitalins just described (dose, $\frac{1}{40}$ to $\frac{1}{20}$ grain). The confusion of these digitalins is apt to lead to dangerous results, and until the question is definitely settled on a basis of scientific exactness, the prescribing of digitalin is not to be advised.

It is of great importance that the pharmacist exercise great care in selecting his stock of digitalis, as the quality ranges from worthless to high potency. The best quality is the English digitalis, and reliable brands of this should be used exclusively by the pharmacist.

A most striking illustration of the variation in quality of digitalis was brought to the notice of the writer by an apothecary, who, on assuming control of a hospital dispensary, threw out the cheap grade digitalis employed by his predecessor, made the tincture from the best English brand, and warned all the house physicians that the excessive doses which they had prescribed of the old tincture were no longer necessary, but were positively dangerous. One of the younger men disregarded his advice, and serious consequences ensued.

The difficulty in selecting digitalis of uniform quality is enhanced by the fact that there is no reliable chemical assay for the drug. Recourse is, therefore, had to a “*physiologic assay*,” and as this is the first drug considered in this book where such assay is used, a few words explanatory of such assays may be of service. Physiologic assays are those in which the activity of the drug is tested upon animals, and in each case where employed the method of procedure varies with the drug under consideration. Thus, digitalis is usually tried on frogs, the amount necessary to stop the heart being taken as the factor. Ergot is given to roosters, the quantity needed to produce a certain purple tint of the comb being the gage of activity. Of course, the tests are far more complex than this scant explanation implies, for example, other factors, such as weight of animal, enter into the calculation leading to standardization. In fact, physiologic testing is beyond the scope of the average pharmaceutical chemist, and for this reason, as well as the fact that the process is not on the same basis of exactness as are chemical assays, the pharmacopœia has not yet provided physiologic assays for official drugs.

The fact that of the several constituents of digitalis given above some are soluble in water and insoluble in alcohol, while with others the solubilities are just reversed, makes it apparent that the composition and medical action of the official *infusion of digitalis* (p. 214) is quite different from that of the official *tincture* (p. 226), *fluidextract* (p. 247), and *extract* (p. 282), the menstruum in the three last mentioned being diluted alcohol. The infusion, containing much digitonin and little digitalin, is a useful diuretic, while the tincture, fluidextract, and extract, containing little digitonin and much digitalin, is a heart tonic. This should plainly show the pharmacist the perniciousness of following the advice of manufacturers by making the infusion from the fluid-

extract—a practice characterized by Hatcher and Sollmann as “absolutely criminal.”

Medical Properties.—A “cardiac tonic,” that is, it slows and strengthens the heart-beat and constricts the blood-vessels. Of peculiar value in dropsy because of its diuretic action.

Dose.—65 milligrammes (1 grain).

LAPPA. Lappa. Burdock.

The dried root of *Arctium Lappa* Linné, or of other species of *Arctium* (Fam. *Compositæ*), collected from plants of the first year's growth.

Constituents.—Supposed to contain a glucoside, of which but little is known, also mucilage and inulin. Note that the roots of plants of the natural order *Compositæ* usually contain inulin (p. 407) instead of starch.

Remarks.—The phrase “from plants of the first year's growth” in the official definition means that the plant, like *digitalis*, is a biennial and that the root is collected in the fall of the first year, when filled with inulin, destined to serve as nourishment for the young shoots of the second year.

Medical Properties.—Supposedly purgative.

Dose.—2 Gm. (30 grains).

JALAPA. Jalap.

The dried tuberous root of *Exogonium Purga* (Wenderoth) Benth (Fam. *Convolvulacæ*), yielding, when assayed by the process given below, not more than 7 per cent. of total resin, of which not more than 15 per cent. should be soluble in ether.

ASSAY OF JALAP

Jalap, in No. 60 powder, ten grammes. 10 Gm.
Ether,
Alcohol,
Chloroform,
Distilled Water, each, a sufficient quantity.

Insert a pledget of purified cotton in the neck of a funnel or small glass percolator, introduce the powdered Jalap and pour ether upon it, keeping the funnel or percolator well covered, until 50 Cc. of percolate have been obtained. Transfer the percolate to a tared beaker, evaporate the ether by means of a water-bath, and weigh the residue. The weight multiplied by ten will give the percentage of ether-soluble resin in the Jalap. Continue the percolation of the powder (which has been exhausted by the ether) with alcohol, until 100 Cc. of percolate have been obtained. Measure 20 Cc. of this percolate into a separator, add 20 Cc. of chloroform, mix the liquids, and then add 20 Cc. of distilled water, and shake the separator thoroughly for one minute. When the liquids have completely separated, draw off the chloroform into a tared beaker, wash the separator with 5 Cc. of chloroform, and add the washings to the tared beaker. Evaporate the chloroform with the aid of a water-bath, and then dry the residue to a constant weight. This weight multiplied by fifty will give the percentage of resin insoluble in ether in the Jalap. Add to this the percentage of ether-soluble resin already determined, and the result will be the percentage of total resin contained in the Jalap.

Constituents.—Resin, consisting chiefly of two glucosides, *jalapin* (ether soluble) and *convolvulin* (insoluble in ether); sugar, starch, etc.

Remarks.—Jalap is a tuberous root, similar to the sweet potato,

and belonging to the same family—the convolvulus or morning-glory family. Note in assay given above that jalap should yield no more than 1.05 per cent. of ether-soluble resin (jalapin) and not less than 5.95 of ether-insoluble resin (convolvulin)—that is, while the drug must yield not less than 7 per cent. total resin, the amount of ether-soluble resin must be not more than 1.05 per cent. This requirement is made because jalapin is nauseating, and it has been recommended that jalap be used only after the jalapin has been removed by percolation with ether. (See p. 1027.)

Medical Properties.—Hydragogue cathartic, that is, a drug producing watery stools. It is administered as *resin* (p. 291), which is a constituent of compound cathartic pills (p. 323), and as *compound powder of jalap* (p. 304), the cream of tartar which the latter contains greatly aiding the action of the jalap.

Dose.—1 Gm. (15 grains).

SCAMMONIUM. Scammony.

A gum-resin obtained by incising the living root of *Convolvulus Scammonia* Linné (Fam. *Convolvulaceæ*).

Constituents.—Resin, consisting chiefly of *scammonin*, gum, etc.

Remarks.—Scammonin is identical with the jalapin from Orizaba jalap (*Ipomœa orizabensis*), but is not the same as the jalapin from official jalap. Fine quality scammony sometimes yields 95 per cent. resin, but usually the quality of the commercial drug is poor; hence the pharmacopœial requirements that 75 per cent. of scammony should dissolve in ether and that there should not be more than 3 per cent. of ash.

Medical Properties.—Hydragogue cathartic. The *resin* (p. 293) is official, and this a constituent of compound extract of colocynth (p. 282).

Dose.—250 milligrammes (4 grains).

COLOCYNTHIS. Colocynth. Bitter Apple.

The peeled dried fruit of *Citrullus Colocynthis* Schrader (Fam. *Cucurbitaceæ*).

Constituents.—*Colocynthin*, a glucoside, resin, etc. No starch.

Remarks.—Colocynth is a fruit of the plant of the gourd family, and closely resembles the common gourd when unpeeled.

Medical Properties.—Hydragogue cathartic. Official preparations, *extract* and *compound extract* (p. 282).

Dose.—65 milligrammes (1 grain).

UVA URSI. Uva Ursi.

The dried leaves of *Arctostaphylos Uva-ursi* (Linné) Sprengel (Fam. *Ericaceæ*).

Constituents.—*Arbutin*, a glucoside (originally called *ursin*), *ericolin*, a glucoside; *ursone*, tannic and gallic acids, resin, gum, etc.

Remarks.—It might be mentioned in passing that the Greek name of the plant (*arctostaphylos*), and the Latin (and specific) name (*uva ursi*) both mean the same thing; namely, *bearberry*, which is the English synonym.

Medical Properties.—Astringent diuretic; antiseptic to the urinary passages. The fluidextract (p. 248) is official.

Dose.—2 Gm. (30 grains).

CHIMAPHILA. *Chimaphila*.

The dried leaves of *Chimaphila umbellata* (Linné) Nuttall (Fam. *Ericaceæ*).

Constituents.—Chimaphilin, $C_{24}H_{21}O_4$; another *glucoside*, $C_{10}H_9O$.

These two bodies are not clearly known, but it may be stated that the constituents of the drug are very similar to uva ursi.

Medical Properties.—Like uva ursi.

Dose.—2 Gm. (30 grains).

QUILLAJA. *Quillaja*. Soap Bark.

The dried bark of *Quillaja Saponaria* Molina (Fam. *Rosaceæ*), deprived of the periderm.

Constituents.—*Saponin*, *sapotoxin*, etc.

Remarks.—Quillaja is the first of the “saponinoid” drugs to be considered—drugs containing saponin, an acrid principle which froths in water like soapsuds. All liquid preparations of such drugs froth when agitated. Soap-bark is used more for washing colored silk (white fabrics are stained brown yellow by its decoctions) than for pharmaceutic purposes. It is, however, used in making emulsions, for which it would be excellent were it not dangerous on account of its irritating properties.

“Gum foam,” used in soda-water syrups, is made by percolating quillaja with water containing glycerin.

Its use raises the same objections as the employment of saponin or of quillaja in emulsions.

Ground soap-bark should be handled with care, as its dust is very irritating to the nostrils, producing violent sneezing. Such drugs are said to be sternutatory.

Medical Properties.—Irritant, rarely used internally.

SARSAPARILLA. *Sarsaparilla*.

The dried root of *Smilax medica* Chamisso and Schlechtendal, *Smilax ornata* Hooker, *Smilax papyracea* Duhamel, or a dried root known commercially as Honduras Sarsaparilla, which is probably obtained from *Smilax officinalis* Kunth (Fam. *Liliacæ*).

Constituents.—*Parillin* (a glucoside similar to saponin, originally called parigin; *saponin*; *sarsasaponin*; starch; calcium oxalate in the form of raphides, etc.

Medical Properties.—*Sarsaparilla* is a drug whose reputation is chiefly false. It is vaunted to the skies by the numerous patent medicine men as the greatest of all blood purifiers, yet cold, calm analysis shows it to have but little merit. It contains saponinoid principles which cause it to froth, and possibly a trace of volatile oil, but neither of these can be regarded as great alteratives, and we are forced to the conclusion that its reputation has been gained by its association with other valuable alteratives. It is rarely prescribed

by itself, its only simple official preparation being the *fluidextract* (p. 247), while its popular preparations are the *compound syrup* (p. 200) and *compound fluidextract* (p. 254), which contain such alteratives as guaiacum and mezereum, and, moreover, these preparations are usually prescribed with potassium iodide.

Dose.—2 Gm. (30 grains).

SENEGA. *Senega*.

The dried root of *Polygala Senega* Linné (Fam. *Polygalaceæ*).

Constituents.—*Senegin* or *polygalic acid* (a form of saponin); an acid also called polygalic acid; trace of volatile oil, pectin, etc. No starch or tannin.

Remarks.—The pectin of senega is a source of annoyance in the pharmaceutical preparations of the drug, these gelatinizing or becoming cloudy on standing, due to conversion of the pectin into the insoluble calcium pectate. This decomposition is prevented by the addition of alkali; hence the senega preparations of 1890 contained ammonia water, while those of U. S. P. VIII. contain potassium hydroxide.

Medical Properties.—Expectorant, nauseant. Emetic in large doses.

Official Preparations.—*Fluidextract* (p. 249), and from this the *syrup* (p. 201) and *compound syrup of squill* (p. 200) are made.

Dose.—1 Gm. (15 grains).

CHAPTER XLI

DERIVATIVES OF THE HIGHER HYDROCARBONS

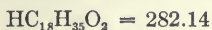
FATS, SOAPS, AND WAXES

As already mentioned, chain hydrocarbons containing more than six carbon atoms are known. Thus we have heptane, C_7H_{16} ; octane, C_8H_{18} ; nonane, C_9H_{20} ; decane, $C_{10}H_{22}$, and likewise the bodies $C_{11}H_{24}$, $C_{12}H_{26}$, and so on to $C_{60}H_{122}$. None of the bodies just mentioned is of interest in pharmacy, save that the petroleum contains variable amounts of almost all the series.

Of pharmaceutical value, however, are the derivatives of $C_{16}H_{34}$ — $C_{18}H_{38}$, $C_{27}H_{56}$, $C_{30}H_{62}$, and of the unsaturated hydrocarbon, $C_{18}H_{36}$.

Hexadecane, $C_{16}H_{34}$, yields a series of alcohols having the formula $C_{16}H_{33}OH$. One of these hexadecyl alcohols is known as *cetyl alcohol* or *æthal*, and is interesting as the alcoholic part of the ester which is the chief constituent of spermaceti. Cetyl alcohol yields on oxidation *palmitic acid*, $C_{15}H_{31}COOH$, which is the common constituent of fats and waxes. Thus the cetin, mentioned above as the chief constituent of spermaceti, is the ester, *cetyl palmitate*.

Likewise the palmitate of glyceryl, commonly known as *palmitin*, is a common constituent of fats.

ACIDUM STEARICUM. Stearic Acid.

A monobasic organic acid [$\text{C}_{17}\text{H}_{35}\text{COOH}$], in its commercial, more or less impure form, usually obtained from the more solid fats, chiefly tallow.

A hard, white, somewhat glossy solid, odorless and tasteless, and permanent in the air.

Insoluble in water; soluble in about 16.6 parts of alcohol at 25° C. (77° F.); readily soluble in boiling alcohol and in ether.

Stearic acid, when pure, melts at 69.2° C. (156.6° F.). The commercial Acid should have a melting-point not lower than 56° C. (132.8° F.), and the melted Acid should become opaque and begin to congeal at a temperature not lower than 54° C. (129.2° F.).

If 1 Gm. of Stearic Acid and 0.5 Gm. of monohydrated sodium carbonate be boiled with 30 Cc. of water, in a capacious flask, the resulting solution, while hot, should not be more than opalescent (limit of *undecomposed fat*).

Tests for Identity.—Given above.

Impurities.—Undecomposed fat. Details above.

Remarks.—Stearic acid is one of the monobasic acids from $\text{C}_{18}\text{H}_{38}$. It is a white, waxy solid, insoluble in water, melting at 70° C. While stearic acid can be made by the reduction of oleic acid, its commercial source is from solid fats, such as tallow and suet, which, on treatment with superheated steam, yield glycerin and stearic acid. Hence most commercial stearic acid is a side-product in glycerin manufacture.

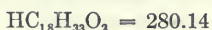
Tallow and suet are composed chiefly of stearin or the stearic acid ester of glycerin, and this, like all esters, can be easily decomposed by treatment with an alkali or with superheated steam by the following reaction:



Stearic acid is official because it enters into the manufacture of glycerin suppositories, a very small quantity of stearic acid soap being ample to solidify a large quantity of glycerin.

Thus in the U. S. P. recipe 60 Gm. glycerin are solidified by heating with 5 Gm. stearic acid and 3 Gm. sodium carbonate, the products of the reaction being glyceryl stearate and sodium stearate.

Margaric acid is a name applied to pearly scales isolated from some fats by Chevreul. Its analysis shows the formula $\text{C}_{16}\text{H}_{33}\text{COOH}$, but careful investigations have shown that this is merely a mixture of palmitic and stearic acids, and in modern chemistry the term finds its last rest in the name applied to artificial butter, *oleo-margarine*.

ACIDUM OLEICUM. Oleic Acid.

A monobasic organic acid, prepared in a sufficiently pure condition by cooling commercial Oleic Acid to about 5° C. (41° F.), then separating and preserving the liquid portion.

A yellowish or brownish-yellow, oily liquid, having a peculiar, lard-like odor and taste; becoming darker and absorbing oxygen on exposure to air.

Specific gravity: about 0.895 at 25° C. (77° F.).

Insoluble in water; soluble in alcohol; chloroform, benzene, petroleum benzin, and fixed and volatile oils.

When cooled to about 4° C. (39.2° F.), Oleic Acid becomes semisolid, and on further cooling congeals to a whitish, solid mass.

When heated to a temperature of about 95°C . (203°F .) decomposition commences, and acrid vapors are produced. At a higher temperature it is completely dissipated.

An alcoholic solution of Oleic Acid has a feebly acid reaction upon blue litmus-paper.

Equal volumes of Oleic Acid and alcohol, mixed at the ordinary temperature, should yield a clear solution without the separation of any oily drops (absence of *fixed oils*).

If 1 Gm. of Oleic Acid be heated with 20 Cc. of alcohol, and 2 drops of phenolphthalein T.S. be added, followed by a strong solution of sodium hydroxide (1 in 4), drop by drop, until the liquid has acquired a permanent red tint and the Acid is saponified, and if acetic acid be added until the red color of the liquid is just discharged, and the liquid be filtered, then 10 Cc. of the filtrate mixed with 10 Cc. of ether should not be rendered more than slightly turbid by shaking with 1 Cc. of lead acetate T.S. (absence of notable quantities of *palmitic* and *stearic acids*).

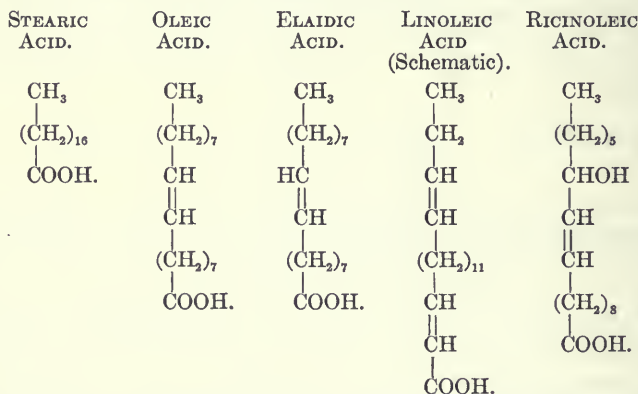
Tests for Identity.—Given above.

Impurities.—Fixed oils, excess of palmitic and stearic acids. Details above.

Remarks.—Oleic acid is obtained from oils by treating same with superheated steam in the process of glycerin manufacture.

It is the product of the unsaturated hydrocarbon $\text{C}_{18}\text{H}_{36}$, and, as can be noticed, it represents stearic acid minus two atoms of hydrogen.

The best comparison of this body with stearic acid and with oleic acid allies can be made by representing the structural formulas side by side:



Oleic acid is a yellow or yellowish-brown, oily liquid, not affecting litmus-paper, and solidifying at 40°C .

On treatment with a large quantity of alcoholic potassium hydroxide it breaks into palmitic and oxalic acid, strong heat with nitric acid breaking it up into a number of simpler bodies; on the other hand, gentle heat with nitrous acid converts it into the isomeric yellow solid *elaidic acid*, the graphic formula of which is given above.

Oleic acid is the chief constituent of liquid fats or oils in the form of the glycerin ester *olein*.

The oleic acid solutions of metallic oxides or alkaloids form the pharmaceutic class, *oleates* (p. 210).

Linolic acid, $\text{C}_{17}\text{H}_{31}\text{COOH}$, differs from oleic acid by possessing two atoms of hydrogen less. It is, therefore, more unsaturated than the oleic. In the form of

glycerin ester it is the chief constituent of linseed oil, the drying properties of which render it so valuable in paint. Most oils spread on wood are absorbed by the fibers leaving a permanent greasy stain; with linseed oil, on the other hand, the stain gradually becomes resinous, due to the oxidation of linolein into the product linoxyn. Very similar to it are the other acids of linseed oil, *linolenic acid* and *isolinolenic acid*. In fact, there still wages a controversy as to whether the last two are not merely impure forms of linoleic acid.

Ricinoleic acid, $C_{17}H_{32}OHCOOH$, represents oleic acid, in which one hydrogen has been replaced by a hydroxyl group. It, in the form of glycerin compound, is the chief constituent of castor oil. Ricinoleic acid forms a chemical combination with sulphuric acid, and the product which is generally obtained by direct treatment of castor oil with sulphuric acid forms with potassa a jelly-like liquid which, on account of its excellent solvent powers, has been called "polysolve." It dissolves a variety of substances, as metallic salts, gums, resins, solid extracts, iodoform, and chrysarobin.

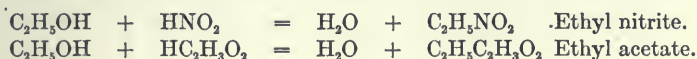
A variety of polysolve has been introduced into American pharmacy under the name *oleite*, as a vehicle for the external application of a variety of medicines.

OILS AND FATS

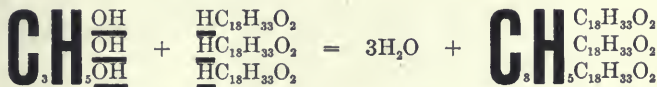
An oil is any liquid which greases—which leaves, when dropped on cloth, a stain which water will not wash out, a stain which makes paper translucent. If a solid substance exhibits similar properties, it is called a fat. Oils are called *volatile* or *fixed*, according as this stain disappears on warming or is permanent by reason of the non-volatility of the oil—characters clearly defined by the names given the two groups.

All fixed oils and fats are combinations of glycerin with oleic, stearic, palmitic, or analogous acids—are glycerin esters of these acids.

As already learned (p. 632), alcohols react with acids, just as do the inorganic basic hydroxides, and such products of alcohols called esters are analogous to the inorganic salts. Among examples of such ester formation already cited were:

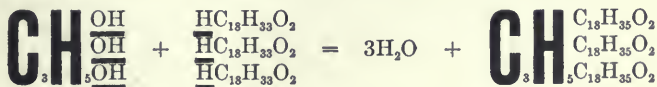


Similarly, glycerin reacts with acids forming esters. Thus, one molecule of glycerin, $C_3H_5(OH)_3$, reacts with three molecules of oleic acid, $HC_{18}H_{33}O_2$, as shown graphically below:



or $C_3H_5(C_{18}H_{33}O_2)_3$, glyceryl oleate or olein.

Similarly, glycerin reacts with stearic acid, $HC_{18}H_{35}O_2$:



or $C_3H_5(C_{18}H_{35}O_2)_3$, glyceryl stearate or stearin.

Combinations of varying proportions of glyceryl oleate (olein), glyceryl palmitate (palmitin), and glyceryl stearate (stearin) form the fixed oils and fats. The larger the proportion of olein, the more liquid

is the fatty substance, oils like that from olives being largely olein. As the amount of stearin increases the more solid the fatty substance becomes, until, in tallow, there are little olein and much stearin.

Fats and fatty oils and waxes are grouped by Allen as follows:

I. Olive oil group: Non-drying vegetable oleins. Examples, olive, almond, peanut, and mustard oils.

II. Cotton seed oil group: Intermediate between I. and III.; hence adulterants of both. Examples: Cotton seed, rape seed, beechnut, and sesame oils.

III. Linseed oil group: Containing linoleic acid, hence drying oils, becoming resinous on boiling with litharge. Examples: Linseed oil and a few others.

IV. Castor oil group: Containing ricinoleic and other acids. Examples: Castor, croton, and Japan wood oils.

V. Palm oil group: Semisolid fats. Examples: Palm, theobroma, and nutmeg oils.

VI. Coconut oil group: Solid esters of lauric acid ($C_{11}H_{23}COOH$). Easily saponified, hence valuable in soap-making. Example: Coconut oil.

VII. Lard oil group: Animal oleins. Examples: Lard, neats-foot, and bone oils.

VIII. Tallow group: Solid animal fats. Examples: Tallow, lard, suint, and butter.

IX. Whale oil group: Marine animal oleins, mixed with glycerides of caproic and valerianic acids. Examples: Whale, porpoise, and cod liver oils.

X. Sperm oil group: Liquid waxes, being esters of monatomic alcohols; thus, sperm oil is the oleate of lauric alcohol, $C_{12}H_{25}OH$. Examples: Sperm, bottlenose, and dolphin oils.

XI. Spermaceti group: Waxes, esters of higher monatomic alcohols. Examples: Spermaceti, beeswax, and Japan wax.

OFFICIAL FIXED OILS AND FATS

OLEUM OLIVÆ. Olive Oil.

A fixed oil expressed from the ripe fruit of *Olea europæa* Linné (Fam. *Oleaceæ*). It should be kept in well-stoppered bottles, in a cool place.

A pale yellow or light greenish-yellow oily liquid, having a slight peculiar odor, and a nut-like, oleaginous taste, with a faintly acid after-taste.

Specific gravity: 0.910 to 0.915 at 25° C. (77° F.).

Very sparingly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulphide.

When cooled to from 8° to 10° C. (46.4° to 50° F.) the Oil becomes somewhat cloudy from the separation of crystalline particles, and at 0° C. (32° F.) it forms a whitish, granular mass.

If 2 Cc. of Olive Oil be shaken vigorously with an equal volume of nitric acid (sp. gr. 1.37), the Oil should retain a light yellow color, not becoming orange or reddish-brown, and after standing for six hours should change into a yellowish-white solid mass and an almost colorless liquid (absence of appreciable quantities of *cotton seed oil* and *most other seed oils*).

If 5 Cc. of the Oil be thoroughly shaken in a test-tube with 5 Cc. of an alcoholic solution of silver nitrate (made by dissolving 0.1 Gm. of silver nitrate in 10 Cc. of alcohol and adding 2 drops of nitric acid), and the mixture be heated for about five minutes in a water-bath, the Oil should retain its original pale color, not becoming reddish or brown, nor should any dark color be produced at the line of contact of the two liquids (absence of more than about 5 per cent. of *cotton seed oil*).

If 2 Cc. of the Oil be mixed in a test-tube with 2 Cc. of equal volumes of amyl alcohol and carbon disulphide containing 1 per cent. of sulphur in solution, and the test-tube be immersed to one-third or one-half its depth in boiling salt water, no reddish color should develop in from ten to fifteen minutes (absence of *cotton seed oil*).

If 2 Cc. of the Oil be mixed with 1 Cc. of hydrochloric acid (sp. gr. 1.18) containing 1 per cent. of sugar, and the mixture be shaken for half a minute and allowed to stand for five minutes, and then 3 Cc. of water added and the whole again shaken, the acid layer should not show a pink color (absence of *sesame oil*).

Tests for Identity.—Given above.

Impurities.—Cotton seed oil, other seed oils, sesame oil. Details above.

Saponification Number.—191 to 195. Details in Part V.

Iodine Absorption Number.—80 to 88. Details in Part V.

Composition.—Olein, 67 per cent.; palmitin, 28 per cent.; linolein, 5 per cent.

Remarks.—Olive oil is obtained by expressing the ripe olive, the same fruit which, when green and pickled, forms the familiar table relish. The oil is rarely found in commerce in pure form.

A very significant fact is that a major part of the cotton seed oil shipped from New Orleans goes to Italy, the home of the olive. For this reason the pharmacopœia provides an elaborate series of tests (given above) for the detection of cotton seed oil. The most reliable of these is the *Halphen test*, the one given above involving the use of amyl alcohol, carbon disulphide, and sulphur.

The last two tests given below—the *saponification number* and the *iodine absorption number*—are of great importance in establishing the constants for all fixed oils.

As, however, a clear understanding of the two tests implies a comprehension of the theory of volumetric analysis, explanation of the tests will be left for the chapter on Pharmaceutical Testing (pp. 1043–1045).

Medical Properties.—Laxative and emollient, externally in liniments.

Dose.—30 Cc. (1 fluidounce).

OLEUM AMYGDALÆ EXPRESSUM. Expressed Oil of Almond.

A fixed oil expressed from Bitter or Sweet Almond. It should be kept in well-stoppered containers, in a cool place.

A clear, pale straw-colored or colorless, oily liquid, almost inodorous, and having a mild, nut-like taste.

Specific gravity: 0.910 to 0.915 at 25° C. (77° F.).

Only slightly soluble in alcohol; soluble in ether, chloroform, and benzene in all proportions.

It should remain clear at –10° C. (14° F.), and it does not congeal until cooled to nearly –20° C. (–4° F.) (absence of *olive oil* or *lard oil*).

If 2 Cc. of the Oil be vigorously shaken with a mixture of 1 Cc. of fuming nitric acid and 1 Cc. of water, a whitish mixture should be formed, which, after standing for some hours at about 10° C. (50° F.), should separate into a solid, white mass and a slightly colored liquid (distinction from the fixed oils of *peach* and *apricot kernels*, which give a red color, and *sesame* and *cotton seed oils*, which are colored brown).

If 10 Cc. of the Oil be mixed with 15 Cc. of a solution of sodium hydroxide (1 in 6) and 10 Cc. of alcohol, and the mixture be allowed to stand at a temperature of 35° to 40° C. (95° to 104° F.), with occasional agitation, until it becomes clear, and if then diluted with 100 Cc. of water, the clear solution thus obtained upon the subsequent addition of an excess of hydrochloric acid will set free a layer of oleic acid. This, when separated from the aqueous liquid, washed with warm water, and clarified by heating on a water-bath, will remain liquid if cooled to 15° C. (59° F.).

One volume of this oleic acid, when mixed with 1 volume of alcohol, should yield a clear solution, which at 15° C. (59° F.) should not deposit any fatty acids nor become turbid upon the further addition of 1 volume of alcohol (distinction from olive, arachis, cotton seed, sesame, and other fixed oils).

Tests for Identity.—Given above.

Impurities.—Olive oil, lard oil, peach kernel, and apricot kernel oils; sesame oil, cotton seed oil, and arachis oil. Details above.

Saponification Number.—191 to 200. Details in Part V.

Iodine Absorption Number.—95 to 100. Details in Part V.

Composition.—Olein, 76 per cent., stearin and palmitin.

Remarks.—The sources of this oil will be studied in other places. Sweet almond in this chapter (p. 735) and bitter almond in Chapter XLIII. (p. 778). A large amount of the commercial oil of sweet almond is, in reality, the fixed oil expressed from peach and apricot kernels, and in the official data given above will be found a test for distinguishing the true oil from these substitutes.

Medical Properties.—Demulcent and laxative. Used in cold cream (p. 345), emulsion of chloroform (p. 270), and emulsion of oil of turpentine (p. 271).

Dose.—30 Cc. (1 fluidounce).

OLEUM GOSSYPII SEMINIS. Cotton Seed Oil.

A fixed oil expressed from the seeds of *Gossypium herbaceum* Linné, or of other species of *Gossypium* (Fam. *Malvaceæ*), and subsequently purified. It should be kept in well-closed containers.

A pale yellow, oily liquid, without odor and having a bland, nut-like taste.

Specific gravity: 0.915 to 0.921 at 25° C. (77° F.).

Very sparingly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulphide.

On cooling the Oil to a temperature below 12° C. (53.6° F.), particles of solid fat will separate. At about 0° to -5° C. (32° to 23° F.) the Oil becomes nearly or quite solid.

If sulphuric acid (specific gravity 1.6 to 1.7) be added to the Oil, preferably diluted with carbon disulphide, a reddish-brown color is rapidly produced.

If 6 Gm. of the Oil be thoroughly shaken in a test-tube for about ten minutes with a mixture of 1.5 Gm. of nitric acid and 0.5 Gm. of water, then heated in a bath of boiling water for not more than fifteen minutes, the Oil will assume an orange or reddish-brown color, and after standing for twelve hours at the ordinary temperature will form a semisolid mass.

If 5 Cc. of the Oil be thoroughly shaken in a test-tube with 5 Cc. of an alcoholic solution of silver nitrate (made by dissolving 0.1 Gm. of silver nitrate in 10 Cc. of alcohol and adding 2 drops of nitric acid), and if the mixture be heated for about five minutes on a water-bath, the Oil will assume a red or reddish-brown color.

If 2 Cc. of the Oil be mixed in a test-tube with 2 Cc. of equal volumes of amyl alcohol and carbon disulphide containing 1 per cent. of sulphur in solution, and the test-tube be immersed to one-third or one-half its depth in boiling salt water, a red color will develop in from ten to fifteen minutes.

Tests for Identity.—Given above.

Saponification Number.—191 to 196. Details in Part V.

Iodine Absorption Number.—102 to 108. Details in Part V.

Remarks.—Cotton seed oil, like all other fixed oils, should be "cold pressed," that is, the expression of the oil from the seed should be done between the cold plates of a hydraulic press (p. 154). In order to increase the output of their product, oil-pressers frequently express the seed between heated plates, in which event the oil contains not

only the liquid fats (olein), but also the solid fats, which are fused by the heated plates. Such an oil is a limpid liquid in warm weather, but on cool days it becomes cloudy and unsightly, the filtration of which is a tedious task.

Any pharmacist who has regard for his reputation will carefully avoid use of such an oil in making official liniment of camphor (p. 273). Whether an oil is cold pressed or hot pressed can be easily tested by subjecting a small vial of it to a cool temperature, say in a refrigerator. It must be borne in mind, however, that the official oil will turn cloudy at a temperature of 12°C .; hence in applying the above test the temperature should not be below 40°C .

The conversion of cotton seed oil to an orange semisolid mass by heating with nitric acid in a water-bath, as given above in one of the official tests, constitutes the *elaidin reaction*. That is, the olein (glyceryl oleate) of the oil is changed into elaidin, the glycerin ester of elaidic acid, described on p. 722. The same reaction takes place with most fixed oils and fats, notably with lard oil and lard, and has pharmaceutical application in the latter case in the manufacture of citrine ointment.

As mentioned on p. 725, cotton seed oil is largely used as an adulterant of olive oil.

As far as health is concerned, it is as good as olive oil, and it seems absurd to pay large prices for it simply because of its trip to Italy and its return in an olive-oil bottle. To do away with this sham as much as possible, the pharmacopeia has directed its use in many preparations formerly calling for olive oil.

Thus it is used in making *liniment of ammonia*—ammonia water 350 Cc., alcohol 50 Cc., cotton seed oil 570 Cc., oleic acid 30 Cc., mix (p. 272) and in *camphorated oil* (p. 273).

Medical Properties.—Demulcent and laxative.

Dose.—16 Cc. (4 fluidrachms).

OLEUM LINI. Linseed Oil.

A fixed oil expressed from Linseed. It should be kept in well-stoppered containers. Linseed Oil which has been "boiled" should not be used nor dispensed.

A yellowish, oily liquid, having a peculiar odor and a bland taste. When exposed to the air, it gradually thickens, darkens in color, and acquires a strong odor and taste; if spread in a thin layer on a glass plate and allowed to stand in a warm place, it is gradually converted into a hard, transparent resin (absence of *non-drying oils*).

Specific gravity: 0.925 to 0.935 at 25°C . (77°F).

It does not congeal at temperatures above -20°C . (-4°F).

It is soluble in about 10 parts of absolute alcohol, and in all proportions in ether, chloroform, petroleum benzin, carbon disulphide, and oil of turpentine.

It should not more than slightly redden blue litmus-paper previously moistened with alcohol (limit of *free acid*).

The Oil should be completely saponifiable with alcoholic potassium hydroxide T.S., and the resulting soap should be completely soluble in water without leaving an oily residue (absence of *mineral oils* and *rosin oil*).

If 2 Cc. of the Oil be warmed and shaken in a test-tube with an equal volume of glacial acetic acid, and if to this mixture, after cooling, 1 drop of sulphuric acid be added, a greenish color should be produced (a violet color under these circumstances indicates the presence of *rosin* or *rosin oils*).

Tests for Identity.—See above.

Impurities.—Non-drying oils, free acids, mineral oils, rosin oil, and rosin. Details above.

Saponification Number.—187 to 195. Details in Part V.

Iodine Absorption Number.—Not less than 170. Details in Part V.

Composition.—Glycerides of linoleic acid, linolenic acid, and isolinolenic acid. (See p. 723.)

Remarks.—The official oil is what is commercially termed “raw oil”; that is, the oil just as it emerges from the press. For painting purposes the raw oil is cooked with litharge, thereby enhancing its “drying” qualities. Such oil is called “boiled oil,” and the pharmacopœial definition given above gives special warning against the use of such boiled oil in medicine. As the boiled oil is more important in commerce than the raw oil, the pharmacist should be sure to specify “raw oil” in buying.

Linseed oil is a “drying oil.” It contains, instead of olein, a similar body, *linolein*, which, however, differs from the former by oxidizing to a solid, *linoxyn*. Hence on being dropped or spread on a board, it eventually hardens into a protective surface, and, accordingly, it and the other drying oils are of value in painting; for which purposes the non-drying oils are unfitted.

It will be noticed among the tests for linseed oil given above that linseed oil is adulterated with mineral oils (various grades of refined petroleum) and with rosin or *rosin oil*; the latter, the product of the destructive distillation of rosin, is rapidly becoming an important article of commerce.

Medical Properties.—Demulcent and laxative. A constituent of *carron oil*—mix equal volumes of linseed oil and lime water (p. 272).

Dose.—30 Cc. (1 fluidounce).

OLEUM RICINI. Castor Oil.

A fixed oil expressed from the seed of *Ricinus communis* Linné (Fam. *Euphorbiaceæ*). It should be kept in well-stoppered containers.

A pale yellowish or almost colorless, transparent, viscid liquid, having a faint, mild odor, and a bland, afterward slightly acid and generally offensive taste.

Specific gravity: 0.945 to 0.965 at 25° C. (77° F.).

Soluble in an equal volume of alcohol, and in all proportions in absolute alcohol or in glacial acetic acid; also soluble, at 25° C. (77° F.), in 3 times its volume of 92.5 per cent. alcohol (absence of more than about 5 per cent. of *most other fixed oils*).

With an equal volume of petroleum benzin it forms at 15° C. (59° F.) a turbid mixture, but at 17° C. (62.6° F.) it yields a clear solution.

When cooled to 0° C. (32° F.) it becomes turbid, with the separation of crystalline flakes, and at about -18° C. (-0.4° F.) it congeals to a yellowish mass.

If 3 Cc. of the Oil be shaken for a few minutes with 3 Cc. of carbon disulphide and 1 Cc. of sulphuric acid, the mixture should not acquire a blackish-brown color (absence of *foreign oils*).

Tests for Identity.—See above.

Impurities.—Other fixed oils. Details above.

Saponification Number.—179 to 183. Details in Part V.

Iodine Absorption Number.—84 to 89. Details in Part V.

Composition.—Note that the olein of this oil is the glyceryl ester of ricinoleic acid (p. 723).

Remarks.—Castor oil is one of the few fixed oils which is freely soluble in official alcohol. For this reason it is useful as the greasy constituent of hair-lotions.

Medical Properties.—Valuable purgative. The only drawback to

the administration of the oil is the disgust it usually excites, hence a disguised form is often demanded. The best method of administration is in sarsaparilla soda-water, the following points being carefully executed. First, put about fifteen drops of compound tincture of cardamom in a clean dry glass, twisting the glass so that its complete interior is moistened with this aromatic. Then add an ounce of regular soda fountain sarsaparilla syrup and squirt in the soda-water with the fine stream. Of course, this will produce a large amount of foam, and at this juncture pour in the dose of oil, carefully avoiding contact with the sides of the glass. Thus poured in, the oil will float between the bottom layer of the sarsaparilla soda and the foam on top, and, as the mixture is swallowed, the foam first and the soda-water last, convey to the tongue and the palate so decided a flavor that the oil slips down almost unnoticed. The compound tincture of cardamom is an essential, as it not only serves to keep the oil from sticking to the glass, but, moreover, by its aromatic odor and taste, does much in disguising the disagreeable odor and taste of the oil.

Dose.—16 Cc. (4 fluidrachms).

The so-called "castor oil capsules"—that is, the small ones of hard gelatin holding about 15 minims—are not to be recommended, as their purgative action is due to substances added to the castor oil, such as croton oil or podophyllin.

OLEUM TIGLII. Croton Oil

A fixed oil expressed from the seeds of *Croton Tiglium* Linné (Fam. *Euphorbiaceæ*). It should be kept in small, well-stoppered bottles, and should be handled with caution.

A pale yellow or brownish-yellow, somewhat viscid, and slightly fluorescent liquid, having a slight fatty odor, and a mild, oily, afterward acrid and burning taste (*great caution is necessary in tasting*).

When applied to the skin it produces rubefaction or a pustular eruption.

Specific gravity: 0.935 to 0.950 at 25° C. (77° F.).

It reddens blue litmus-paper moistened with alcohol.

When fresh it is soluble in from 55 to 60 parts of alcohol, the solubility increasing by age; freely soluble in ether, chloroform, carbon disulphide, and in fixed or volatile oils.

When gently heated with twice its volume of absolute alcohol, it forms a clear solution from which the Croton Oil should separate on cooling.

If to 2 Cc. of Croton Oil 1 Cc. of fuming nitric acid and 1 Cc. of water be added, and the mixture vigorously shaken, it should not solidify either completely or partially, after standing for one or two days (absence of *other non-drying oils*).

Tests for Identity.—Details above.

Impurities.—Other non-drying oils. Details above.

Saponification Number.—203–215. Details in Part V.

Iodine Absorption Number.—103–109. Details in Part V.

Composition.—Glycerides of tiglinic (methyl-crotonic), acetic, butyric, valerianic, stearic, palmitic, laurinic, myristic, crotonic, oleic, and formic acids. A resin-like blistering substance, *crotonol*, $C_{19}H_{34}O_2$.

Medical Properties.—A powerful purgative, used only in cases of last resort, when all other purgatives have failed, its action being too violent for general use. It is a vesicant, producing blisters on application to the skin.

It is usually administered internally in the form of pills, the oil being solidified by use of crumb of bread.

Dose.— $\frac{1}{20}$ Cc. (1 minim).

OLEUM THEOBROMATIS. Oil of Theobroma.

A fixed oil expressed from the roasted seeds of *Theobroma Cacao* Linné (Fam. *Sterculiaceæ*).

A yellowish-white solid, having a faint, agreeable odor, and a bland, chocolate-like taste.

Specific gravity: 0.970 to 0.976 at 25° C. (77° F.).

Oil of Theobroma should be brittle at temperatures below 15° C. (59° F.), and should melt at 30° to 35° C. (86° to 95° F.) to a clear liquid.

It is readily soluble in ether, chloroform, or benzene; also soluble in 100 parts of cold absolute alcohol and in 20 parts of boiling absolute alcohol; the solutions should be neutral to test-paper.

If 1 Gm. of Oil of Theobroma be dissolved in 3 Cc. of ether in a test-tube at a temperature of 17° C. (62.6° F.), and the tube frequently plunged into water at 0° C. (32° F.), the liquid should not become turbid nor deposit white flakes in less than three minutes; and if the mixture after congealing be again brought to 15° C. (59° F.), it should gradually form a perfectly clear liquid (absence of *wax*, *stearin*, *tallow*, etc.).

Tests for Identity.—Details above.

Impurities.—Wax, stearin, tallow. Details given above.

Saponification Number.—188 to 195. Details in Part V.

Iodine Absorption Number.—33 to 38. Details in Part V.

Composition.—Glycerides of oleic, lauric, palmitic, stearic, and arachic acids.

Remarks.—Oil of theobroma is, chemically and physically, just between the oils and fats. While a hard solid at ordinary temperatures, the heat of the body is sufficient to melt it—to convert it to a liquid. This valuable property suggested to Mr. A. B. Taylor its use as a basis for suppositories, every other fat melting at so low a temperature being a soft, sticky mass at ordinary temperatures.

Note that the common term “cocoa-butter” is replaced in the pharmacopœia by the more accurate “butter of cacao” (the latter word being pronounced as if spelled “ka-ka-o”). This step relieves the confusion arising from the fact that three different plants have been given very similar names. These plants and products are:

Cacao (cocoa) and butter of cacao from *Theobroma Cacao*, the chocolate plant.

Coconut, from *Cocis nucifera*, the coconut palm.

Coca-leaves, from *Erythroxylon Coca*, the coca shrub.

Hence the necessity of learning to say “cacao,” for otherwise one is sure to confound the plant yielding chocolate and oil of theobroma with the coconut palm or with the coca tree.

Medical Uses.—Suppository base. Emollient for chapped hands.

OLEUM ADIPIS. Lard Oil.

A fixed oil expressed from lard at a low temperature.

A colorless or pale yellow, oily liquid, having a peculiar odor and a bland taste.

Specific gravity: 0.905 to 0.915 at 25° C. (77° F.).

At a temperature a little below 10° C. (50° F.) it usually commences to deposit a white, granular fat, and at or near 0° C. (32° F.) it forms a solid white mass.

Lard oil is almost insoluble in cold alcohol and only slightly soluble in boiling alcohol; easily soluble in ether, chloroform, benzene, and carbon disulphide.

If 5 Cc. of the Oil be thoroughly shaken in a test-tube with 5 Cc. of an alcoholic solution of silver nitrate (made by dissolving 0.1 Gm. of silver nitrate in 10 Cc. of alcohol, and adding 2 drops of nitric acid), and the mixture heated for about five minutes on a water-bath, the Oil should remain nearly or quite colorless, not acquiring a reddish or brown color (absence of more than about 5 per cent. of *cotton seed oil*).

Tests for Identity.—Given above.

Impurities.—Cotton seed oil (silver nitrate test and amyl-alcohol-sulphur test given under Olive Oil), mineral oils (saponification test given under Linseed Oil).

Saponification Number.—195 to 197. Details in Part V.

Iodine Absorption Number.—56 to 74. Details in Part V.

Composition.—Olein, some linolein, stearin and palmitin, the amount of these solids varying from very little in the best "winter strained oil," to large amounts when oil has been carelessly pressed.

Remarks.—Note that the pharmacopœia specifies that this oil be expressed from lard at a low temperature. Instead of calling it, as in the case of castor oil, "cold pressed," this cold pressed lard oil is called in commerce "*winter strained*," the two terms meaning practically the same thing.

Lard oil, like linseed oil, is largely adulterated with various petroleum oils; hence the pharmacopœial test for mineral oils. It was chiefly the presence of these mineral oils that made difficult the manufacture of citrine ointment from lard oil, as directed by U. S. P. 1890, and for this reason the ointment of U. S. P. VIII. is made from lard.

Medical Properties.—Emollient. Used chiefly as ointment base.

OLEUM MORRHUÆ. Cod Liver Oil.

A fixed oil obtained from the fresh livers of *Gadus morrhua* Linné, and of other species of *Gadus*. It should be kept in a cool place, in well-stoppered bottles, which have been thoroughly dried before filling.

A pale yellow, thin, oily liquid, having a peculiar, slightly fishy, but not rancid odor, and a bland, fishy taste.

Specific gravity: 0.918 to 0.922 at 25° C. (77° F.).

Very slightly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulphide; also in 2.5 parts of acetic ether.

If 1 drop of the Oil be dissolved in 20 drops of chloroform and the solution shaken with 1 drop of sulphuric acid, the solution will acquire a violet-red tint, rapidly changing to rose-red and, finally, brownish-yellow.

If a glass rod moistened with sulphuric acid be drawn through a few drops of the Oil, on a porcelain plate, a violet color will be produced.

Cod Liver Oil should be only very slightly acid to blue litmus-paper which has been previously moistened with alcohol (limit of *free fatty acids*).

If 2 or 3 drops of fuming nitric acid be allowed to flow alongside of 10 or 15 drops of the Oil, contained in a watch-glass, a red color will be produced at the point of contact. On stirring the mixture with a glass rod, this color becomes bright rose-red, soon changing to lemon-yellow (distinction from *seal oil*, which shows at first no change of color, and from *other fish oils*, which become at first blue and afterward brown and yellow).

Tests for Identity.—Impurities, free fatty acids, seal oil, and other fish oils.

Saponification Number.—175 to 185. Details in Part V.

Iodine Absorption Number.—140 to 150. Details in Part V.

Manufacture.—The oil is separated from fresh livers either by exposing to sun, when oil exudes; by slowly heating on steam bath; or by boiling with water and skimming off the separated oil.

Composition.—Olein, little palmitin and stearin, traces bromides and iodides, cholesterin, and some animal decomposition products (alkaloids).

Remarks.—The best grade of cod liver oil is that obtained from the fisheries on the coast of Norway, although some grades of Newfoundland oil are of fair quality. The commercial oil is frequently adulterated with other fish oils; hence the nitric acid color test provided by the pharmacopœia. The keeping of cod liver oil deserves more care than is usually bestowed upon it by the pharmacist, hence the pharmacopœial injunction as to storing should be followed unless the druggist is willing to dispense a rankly smelling and tasting, partly decomposed product.

The efforts of some manufacturers to dispense a cod liver oil product minus the oil, such as the numerous wines of cod liver oil, is an example of the play Hamlet with the hero Hamlet omitted; for it is now considered by the leading therapeutists that the entire medicinal value of the oil is because of its nutritive value, and that the so-called "active principles," variously called *gadulol*, *morrhual*, etc., are worse than useless. These substances, usually extracts prepared by shaking the oil with alcohol, separating the alcoholic layer, and distilling off the solvent, when redissolved in aromatized alcoholic liquors, form the elixirs and wines of cod liver oil.

Medical Properties.—Demulcent, nutritive in wasting diseases. Best administered as emulsions (p. 271). Of these, *emulsion of cod liver oil* and *emulsion of cod liver oil with hypophosphites* are official.

Dose.—16 Cc. (4 fluidrachms).

ADEPS. Lard.

The prepared internal fat of the abdomen of the hog (*Sus scrofa*, var. *domesticus* Gray), purified by washing, melting, and straining. It should be kept in well-closed vessels impervious to fat, in a cool place.

A soft, white, unctuous solid, having a faint odor free from rancidity, and a bland taste.

Insoluble in water; very slightly soluble in alcohol; readily soluble in ether, chloroform, carbon disulphide, or petroleum benzin.

Specific gravity: about 0.917 at 25° C. (77° F.) and about 0.904 at 40° C. (104° F.), water at 25° C. (77° F.) taken as the standard.

It melts at 38° to 40° C. (100.4° to 104° F.) to a perfectly clear liquid, which is colorless in thin layers and from which an aqueous layer should not separate.

Distilled water boiled with Lard should not acquire an alkaline reaction (absence of *alkalis*). A portion of the water, when filtered, acidulated with nitric acid and treated with silver nitrate T.S., should not yield a white precipitate soluble in ammonia water (absence of *chlorides*).

If 10 Gm. of Lard be dissolved in chloroform, and the solution mixed with 10 Cc. of alcohol and 1 drop of phenolphthalein T.S., it should not require more than 0.2 Cc. of normal potassium hydroxide V.S. to produce a pink tint after strongly shaking (limit of *free fatty acids*).

Tests for Identity.—Given above.

Impurities.—Alkalis, chlorides, excess of fatty acids. Details above. Cotton seed oil. (Haliphen and silver nitrate tests given under Olive Oil.)

Composition.—Olein (lard oil), 60 per cent.; palmitin and stearin, 40 per cent.

Remarks.—Note that, according to the rule laid down when discussing fats (p. 723), whether a fat is liquid or solid depends on the

relative amount of olein and stearin it contains. Olive oil is almost pure olein; suet, which is hard, is almost pure stearin, while lard, which is half-and-half, is semisolid.

The solid fat of cotton seed oil is used for adulterating lard, and the pharmacopœial test for this impurity is given above.

Medical Properties.—Emollient and ointment base. Now used instead of lard oil in making citrine ointment (p. 620).

ADEPS BENZOINATUS. Benzoinated Lard.

Recipe.—Lard, one thousand grammes..... 1000 Gm.
Benzoin, in coarse powder, twenty grammes..... 20 Gm.

Add the Benzoin to the Lard and mix thoroughly; then melt the Lard by means of a water-bath, and, stirring frequently, continue the heat for two hours, covering the vessel and not allowing the temperature to rise above 60° C. (140° F.). Lastly, strain the liquid through muslin and stir occasionally while it cools.

When Benzoinated Lard is to be kept or used during warm weather, 5 per cent. (or more, if necessary) of the Lard should be replaced by White Wax.

Remarks.—It will be seen that this official is made by digesting ground benzoin in melted lard, and *not* by mixing lard with tincture of benzoin, as the writer has known some careless druggists to do. Lard rapidly turns rancid, and the benzoin is added to hinder the decomposition.

SEVUM PRÆPARATUM. Prepared Suet.

[SEVUM, PHARM. 1890]

The internal fat of the abdomen of the sheep *Ovis aries* Linné, purified by melting and straining.

Prepared Suet should be kept in well-closed vessels impervious to fat. It should not be used after it has become rancid.

A white, solid fat, nearly inodorous, and having a bland taste when fresh, but becoming rancid on prolonged exposure to the air.

Insoluble in water or cold alcohol; soluble in 44 parts of boiling alcohol, in about 60 parts of ether, and slowly in 2 parts of petroleum benzin. From its solution in the latter, when kept in a stoppered flask, it slowly separates in a crystalline form on standing.

An alcoholic solution of Prepared Suet is neutral or has only a slight acid reaction upon litmus-paper moistened with alcohol.

Prepared Suet melts between 45° and 50° C. (113° and 122° F.), and congeals between 37° and 40° C. (98.6° and 104° F.).

Composition.—Stearin and palmitin, 70 per cent.; olein, 30 per cent.; hercin, a trace.

Remarks.—Suet turns rancid even more rapidly than lard. Borrell found that when melted suet was digested with elm bark the resulting "*ulmated suet*" kept quite well.

Medical Properties.—Used for thickening cerates.

ADEPS LANÆ. Wool-Fat.

The purified fat of the wool of sheep (*Ovis aries* Linné) freed from water.

A light yellowish, tenacious, unctuous mass, having a slight, peculiar odor.

Insoluble in, but miscible with, large quantities of water, sparingly soluble in cold alcohol, more soluble in hot alcohol, readily soluble in ether and chloroform.

Wool-Fat melts at about 40° C. (104° F.), and at a higher temperature vaporizes, the vapor igniting and burning with a luminous, sooty flame.

The solution of Wool-Fat in chloroform (1 in 50), when poured upon the surface of concentrated sulphuric acid, gradually develops a deep brownish-red color at the line of contact of the layers.

When Wool-Fat is incinerated it should leave not more than 0.3 per cent. of ash, which should not show an alkaline reaction to litmus-paper (freedom from *alkalis*).

If 2 Gm. of Wool-Fat be dissolved in 10 Cc. of ether, and 2 drops of phenolphthalein T.S. added, a colorless liquid should be obtained (absence of *free alkalis*), which, on the addition of 1 drop of normal potassium hydroxide V.S., should develop a deep red color (absence of *free fatty acids*).

If 1 Gm. of Wool-Fat be boiled with 20 Cc. of alcohol, and the solution filtered after cooling, the filtrate should not be rendered turbid by the addition of an alcoholic solution of silver nitrate (1 in 20) (absence of *chlorides*).

If 10 Gm. of Wool-Fat be heated with 50 Cc. of water on a bath of boiling water until completely fused, the lower aqueous layer, when filtered through a well-wetted filter, should not yield *glycerin* on evaporation, and when boiled with potassium hydroxide T.S. it should not give off vapors of ammonia (absence of *organic nitrogenous matter*).

Tests for Identity.—Given above.

Impurities.—Alkalis, free fatty acids, chlorides, glycerin, and organic nitrogenous matter. Details above.

Manufacture.—Crude suint (the alkaline washings from sheep-wool) is treated with acid, thereby liberating the fatty acid combination of cholesterolin and isocholesterin, and also 30 per cent. free fatty acid. The fatty mixture is then treated with caustic alkali, which saponifies the free fats and emulsifies the cholesterolin compounds. The mixture is centrifuged, when the cholesterolin compounds separate as cream and are thus removed from bad-smelling fatty acids.

Composition.—A mixture of the esters of oleic, stearic, palmitic, cerotic, caproic, butyric, and isovaleric acids with the cholesterolins and some ceryl alcohol. It contains no glyceryl compounds, as do most fats.

Remarks.—The fat of sheep-wool has already been considered as *suint*, under the head of the Potassium Salts (p. 431).

Purified suint was introduced in modern medicine under the copyrighted name *lanolin*, and since its introduction has enjoyed great popularity, because of the fact that it is more readily absorbed by the pores of the skin than lard or other fats.

It readily absorbs water, and when combined with 30 per cent. of that fluid, it forms the official *hydrous wool-fat*.

ADEPS LANÆ HYDROSUS. Hydrous Wool-fat.

The purified fat of the wool of sheep (*Ovis aries* Linné), mixed with not more than 30 per cent. of water.

A yellowish-white, or nearly white, ointment-like mass, having a slight, peculiar odor.

Insoluble in water, but miscible with twice its weight of the latter, without losing its ointment-like character. With ether or chloroform it yields turbid solutions which are neutral to moistened litmus-paper.

Hydrous Wool-Fat melts at about 40° C. (104° F.), and separates into an upper oily and a lower aqueous layer. When heated on a water-bath, with stirring until it ceases to lose weight, there should remain not less than 70 per cent. of a residue, which is transparent when melted, and when cold remains as a yellowish, tenacious, unctuous mass, completely soluble in ether or chloroform, and only sparingly soluble in alcohol. If Hydrous Wool-Fat be thus deprived of water, it should respond to the tests given under *Adeps Lanæ*.

Manufacture.—Made by melting wool-fat, gradually adding 30 per cent. warm water, and triturating until cool.

Medical Properties.—Emollient and ointment base. Used in the following official ointments: *Belladonna*, *ammoniated mercury*, *yellow mercuric oxide*, *red mercuric oxide*, and *stramonium*.

DRUGS CONTAINING FIXED OILS

Fixed oils are usually found in the seed of plants, although there are some exceptions. Thus butternut bark (*juglans*, U. S. P. 1890) contains appreciable quantities of fixed oil. In many official seeds, such as *strophanthus* (p. 714), the fixed oil is of importance only because its presence is a source of annoyance in making pharmaceutical preparations; hence in this chapter we will discuss only those drugs that owe their entire therapeutic value to the fixed oil they contain.

The **mustards** (white or black) will be considered under the sulphocyanates (p. 746), since their activity is due to their sulphurated volatile oils.

Bitter almond will be considered under *benzaldehyde*, since its active principle, the volatile oil, is composed chiefly of that body.

AMYGDALA DULCIS. Sweet Almond.

The ripe seed of *Prunus Amygdalus* Stokes, var. *dulcis* De Candolle (Fam. *Rosaceæ*).

Constituents.—Fixed oil, 50 per cent. (expressed oil of almond, U. S. P.); albumin, 25 per cent.; the ferment emulsin; asparagin. No amygdalin.

Remarks.—Since sweet almond contains no amygdalin, no volatile oil nor hydrocyanic acid odor is produced on moistening the seed, even though the ferment, emulsin, is present. Almonds are *blanched* by removing the scurfy seed-coat. This is accomplished by immersing them in hot water for a few minutes, when the coat can be easily slipped off.

Medical Properties.—Demulcent. Used in *emulsion of sweet almond*, U. S. P. (p. 268).

LINUM. Linseed. Flaxseed.

The ripe seed of *Linum usitatissimum* Linné (Fam. *Linaceæ*).

Ground Linseed (Linseed Meal or Flaxseed Meal) should be recently prepared and free from unpleasant or rancid odor. It is a grayish-yellow powder containing brownish fragments, and when exhausted by carbon disulphide should yield not less than 30 per cent. of a fixed oil, all of which is saponifiable.

If 0.1 Gm. of ground Linseed be mixed with 20 Cc. of water and the mixture heated to boiling, then cooled and diluted with cold water to 100 Cc., the addition of 0.5 Cc. of iodine T.S. should not produce more than a pale blue color (limit of *starch*).

Constituents.—Fixed oil, 30 per cent.; (*oleum lini*, U. S. P.) mucilage, 15 per cent. ($C_{12}H_{20}O_{10}$); albumin; linamarin (like amygdalin), etc.

Remarks.—It is difficult to decide whether flaxseed should be grouped among the mucilaginous or the oleaginous drugs.

The whole seed is used entirely, because of the mucilage, while in the ground seed both the oil and the mucilage play a therapeutic rôle. The mucilage of the seed is interesting, as being found entirely in the outer layer of cells of the testa; hence by immersing the whole seed in water all the mucilage is extracted.

Much of the ground flaxseed of commerce is the seed deprived of part or all of its oil; hence the pharmacopœial test given above requiring at least 30 per cent. of oil.

Linseed contains some starch, but not large amounts, and thus differs from most of the adulterants of the ground seed. Above is given the pharmacopœial test for "limit of starch," the test having been devised by Professor J. U. Lloyd.

Medical Properties.—The mucilage (hence the unground seed) is demulcent. Therefore an infusion of the whole seed is used as a basis of domestic cough-mixtures, while a thicker solution is sometimes used as a bandolin or as a face jelly, although for this purpose quince seed or tragacanth is more useful. Ground seed mixed with hot water to thick paste forms the familiar "*flaxseed poultice*."

PEPO. Pepo. Pumpkin Seed.

The ripe seed of *Cucurbita Pepo* Linné (Fam. *Cucurbitaceæ*).

Constituent.—Fixed oil, 20 to 40 per cent., sugar, starch. Resin of tenifuge properties.

Medical Properties.—Tenifuge—expeller of tape-worm.

Dose.—30 Gm. (1 ounce).

LYCOPodium. Lycopodium.

The spores of *Lycopodium clavatum* Linné or of other species of *Lycopodium* (Fam. *Lycopodiaceæ*).

Constituents.—Fixed oil, 50 per cent.

Remarks.—The official definition of lycopodium is "the spores of *Lycopodium clavatum*," etc. The spores of non-flowering plants are organs midway between the fruit and the seed of flowering species, though there is a decided difference in the way the two sprout into new plants.

The large quantity of oil contained in lycopodium accounts for its mobility—its smooth, slippery character—as well as causing the remarkably quick combustion of lycopodium when blown into a flame. It is very difficult to extract the oil from lycopodium, as it is next to impossible completely to break the walls of the individual cell which forms each sporule. By long contusion with sand the result may be reached.

Because of its mobility it is called "*witch-powder*" in some sections of the country.

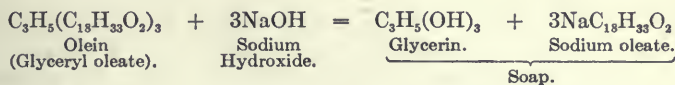
Medical Properties.—Externally, as a dusting-powder. Its use as a dusting-powder for pills has been already given.

SOAPS

Soaps are products resulting from the action of sodium and potassium hydroxide on oils or fats. All the ingredients employed in commercial soap-making are more or less impure, hence the products are not definite chemical compounds.

The pharmacopœia recognizes as soap—*sapo*—made from olive oil and soda—and *sapo mollis*—from linseed oil and potassa. Fixed

oils are combinations of the oleate, stearate, and palmitate of glyceryl. If we take glyceryl oleate and treat it with sodium hydroxide we get a reaction quite similar to that occurring when fat is treated with steam (p. 680), namely:



SAPON. Soap.

Soap prepared from sodium hydroxide and olive oil.

A white or whitish solid, hard, yet easily cut when fresh, having a faint, peculiar odor free from rancidity, a disagreeable, alkaline taste, and an alkaline reaction.

Soluble in water and in alcohol, more readily with the aid of heat.

On placing 0.5 Gm. of Soap, together with about 10 Cc. of alcohol, in a tared beaker containing 1 Gm. of dry, clean sand, evaporating the resulting solution of the Soap to dryness, and drying the residue at 110° C. (230° F.), to a constant weight, the loss should not exceed 36 per cent. (absence of an undue amount of water).

An alcoholic solution of Soap (1 in 25) should not gelatinize on cooling (absence of animal fats).

An aqueous solution of Soap (1 in 20) should remain unchanged in color upon the addition of ammonium sulphide T.S.; and upon acidulating another portion of the solution with hydrochloric acid and filtering, the filtrate should remain unchanged in color when an equal volume of hydrogen sulphide T.S. is added and the mixture is allowed to stand well stoppered in a warm place for half an hour (absence of metallic impurities).

On dissolving 20 Gm. of Soap in alcohol, with the aid of heat, transferring the undissolved residue, if any, to a tared filter, and washing it thoroughly with boiling alcohol, it should, after drying, weigh not more than 0.8 Gm. (limit of sodium carbonate, etc.); of this residue not more than 0.2 Gm. should be insoluble in water (limit of silica and other accidental impurities).

If a solution of 5 Gm. of Soap in 50 Cc. of hot water be mixed with 3 Cc. of tenth-normal oxalic acid V.S., the subsequent addition of a few drops of phenolphthalein T.S. should produce no pink or red tint (limit of alkalinity).

Tests for Identity.—See above.

Impurities.—Excess of water, animal fats, metallic impurities. Sodium carbonate, silica, excess of alkalinity. Details above.

Remarks.—Soap is a product of the treatment of an oil, supposedly olive oil, with sodium hydroxide. This is called "Castile soap," because originally made in the city of Castile, Spain, and at the present time the finest brand of Castile soap on the market is the "Yaritu" brand, manufactured in that city. The large majority of the so-called Castile soap, however, is manufactured in Marseilles.

Soap is a hard, whitish solid, insoluble in salt water, very soluble in alcohol, and somewhat soluble in cold water. When a solution of soap is made with excess of water, the soap breaks into free alkali and fatty acids. The alkali immediately attacks the skin if brought in contact with it, but before it can do much harm the fatty acid acts as a soothing agent. This explains the whole plan of washing with soap, the alkali removing grease and at the same time removing minute traces of skin, while the fatty acids prevent the burning which invariably results if the free alkali were used for the same purpose.

As seen above, Castile soap is a mixture of glycerin and sodium oleate. The red Castile soap is colored by the addition of ferrous oxide, which is black, and this, on exposure to air, is oxidized to red ferric oxide—iron rust. The effect can be noticed by making a fresh

cut through a cake of mottled Castile soap, which shows on the interior black streakings which, on exposure to air, gradually assume the red hue of the rest of the soap. The coloring (or mottling) is simply to please the eye, as the ferric oxide is of no influence, save to increase weight.

Soap is used chiefly externally, although it is sometimes administered internally as a laxative, especially in purgative pills. For this purpose it is used in the following official pills: *aloes*, *asafetida*, *opium*. It is also a constituent of *compound extract of colocynth*, and thus as the excipient in *compound cathartic pills* and *vegetable cathartic pills* (p. 323).

An alcoholic solution of soap containing camphor and oil of rosemary is official as "soap liniment" (p. 273).

SAPO MOLLIS. Soft Soap.

<i>Recipe</i> .—Linseed Oil, four hundred grammes.....	400 Gm.
Potassium Hydroxide, ninety-five grammes.....	95 Gm.
Alcohol, forty cubic centimeters.....	40 Gm.
Water, a sufficient quantity.	

Heat the Linseed Oil in a deep, capacious vessel, on a water-bath or steam-bath, to a temperature of about 70° C. (158° F.). Dissolve the Potassium Hydroxide in four hundred and fifty cubic centimeters of Water, warm the solution to about 70° C. (158° F.), add it to the Linseed Oil, and mix thoroughly; then incorporate the Alcohol and continue the heat (without stirring) until a small portion of the mixture is found to be soluble in boiling Water without the separation of oily drops. Then allow the mixture to cool and transfer it to suitable vessels.

The Potassium Hydroxide used in this process should be of the full strength directed in the Pharmacopœia (85 per cent.). Potassium Hydroxide of any other strength, however, may be used, if a proportionately larger or smaller quantity be taken, the proper amount for the above formula being ascertained by dividing 8075 by the percentage of absolute Potassium Hydroxide contained therein.

A soft, unctuous, yellowish-brown mass, having a characteristic odor and an alkaline taste.

An aqueous solution shows an alkaline reaction to red litmus-paper.

If to a solution of 5 Gm. of Soft Soap in 50 Cc. of water, 2 drops of phenolphthalein T.S. be added, not less than 2.3 Cc. nor more than 4.5 Cc. of tenth-normal oxalic acid V.S. should be required to discharge the red tint (limit of *free alkali*).

Soluble in hot water to a nearly clear liquid; also in hot alcohol without leaving more than 3 per cent. of insoluble residue.

Recipe.—Given above.

Tests for Identity.—Given above.

Impurity.—Excess of free alkali, given above.

Soft soap is a soap made by treatment of linseed oil with potassa, and clearing the product with an appropriate quantity of alcohol. *Green soap* is a soft soap made with potassa and greenish rape seed oil.

The two soaps (hard and soft) are interesting as illustrating the different actions of the salts of two metals. The sodium salts are usually efflorescent and, similarly, Castile soap is found to effloresce on standing. The potassium compounds, like potassa soap, are deliquescent.

Soft soap is used externally in skin troubles, and is a constituent of the official liniment of soft soap (p. 274).

While Castile soap and soft soap are the only official soaps, in the limited sense of the term, we have other official products, which can

be considered as soaps, if the definition is extended to all metallic combinations of the acids found in fats.

Thus *liniment of ammonia*, made with cotton seed oil and ammonia water, is an ammonia soap; *liniment of lime*, made with linseed oil and lime water, is a lime soap; *liniment of lead subacetate* (U. S. P. 1880) is one variety of lead soap, while *lead plaster* is another. The three last mentioned are examples of soaps insoluble in water.

The official *oleates* are scarcely to be considered as soaps, as they all contain a large excess of oleic acid.

In most works on pharmacy the chapter on fats and soaps is considered the proper place to consider the petroleum products, but these have already been described on p. 636, for reasons mentioned there; hence at this place all that is necessary is the mention that animal and vegetable fats being the oleates, stearates, and palmitates of glyceryl, or closely allied bodies, when treated with sodium or potassium hydroxide, split into glycerin and soap; in other words, can be saponified. The mineral fats—petrolatum and similar products—do not saponify when treated with sodium or potassium hydroxide. Again, animal and vegetable fats, on standing, become oxidized, partially splitting into glycerin and the fatty acids, becoming rancid, as we say.

Petroleum fats do not turn rancid, do not oxidize, hence ointments made with vaseline and similar petroleum fats keep indefinitely. Petroleum, however, is not absorbed by the skin as readily as are the animal and vegetable fats.

WAXES

Among the derivatives of the fatty hydrocarbons higher than $C_{18}H_{38}$ some bodies of pharmaceutic interest are found. $C_{27}H_{56}$ yields ceryl alcohol or *cerotin* ($C_{27}H_{55}OH$), while the body, $C_{30}H_{61}OH$ or $C_{31}H_{63}OH$, is known as melissyl alcohol (sometimes called *myricyl alcohol*).

The ultimate analysis of these bodies containing a large number of carbon atoms does not enable us accurately to determine the formula. The difference in the percentage of carbon and hydrogen in the bodies $C_{30}H_{61}OH$ and $C_{31}H_{63}OH$ varies so slightly as to give rise to doubt as to which is the exact formula.

Thus, $C_{30}H_{61}OH$ has the molecular weight:

$$\begin{array}{rcl} C_{30} & = & 30 \times 12 = 360 \\ H_{62} & = & 62 \times 1 = 62 \\ O & = & 1 \times 16 = 16 \end{array}$$

molecular weight, 438, and contains

$$\begin{array}{l} \text{Carbon, } \frac{360}{438} \times 100 = 82.19 \text{ per cent.} \\ \text{Hydrogen, } \frac{62}{438} \times 100 = 14.15 \text{ per cent.} \\ \text{Oxygen, } \frac{16}{438} \times 100 = \frac{3.65}{99.99} \text{ per cent.} \end{array}$$

While $C_{31}H_{63}OH$ has the molecular weight:

$$\begin{array}{rcl} C_{30} & = & 31 \times 12 = 372 \\ H_{64} & = & 64 \times 1 = 64 \\ O & = & 1 \times 16 = 16 \end{array}$$

molecular weight, 452, and contains

$$\begin{array}{l} \text{Carbon, } \frac{372}{452} \times 100 = 82.30 \text{ per cent.} \\ \text{Hydrogen, } \frac{64}{452} \times 100 = 14.15 \text{ per cent.} \\ \text{Oxygen, } \frac{16}{452} \times 100 = \frac{3.54}{99.99} \text{ per cent.} \end{array}$$

The difference in percentage of carbon in these substances (82.19 per cent. and 82.30 per cent.) is no greater than the permissible limit of error in analysis; while the percentage of hydrogen in the two is identical.

The oxidation of ceryl alcohol yields *cerotinic acid*, $C_{26}H_{53}COOH$, while the oxidation of melissyl alcohol gives *melissic acid*, $C_{29}H_{59}COOH$ or $C_{30}H_{61}COOH$.

These four bodies are of interest as constituents of the various waxes, they being esters of the two alcohols, with the two acids just mentioned and other fatty acids.

CERA FLAVA. Yellow Wax.

A solid substance prepared from the honeycomb of the bee, *Apis mellifera* Linné.

A yellowish to brownish-yellow solid, having an agreeable, honey-like odor, and a faint balsamic taste.

Specific gravity: 0.951 to 0.960 at 25° C. (77° F.).

Melting-point: 62° to 64° C. (143.6° to 147.2° F.).

It is somewhat brittle when cold, and when broken presents a dull, granular, not crystalline fracture. By the heat of the hand it becomes plastic.

Yellow Wax is insoluble in water, sparingly soluble in cold alcohol; boiling alcohol dissolves the cerotic acid and a portion of the myricin. It is completely soluble in ether, chloroform, and in fixed and volatile oils; partially soluble in cold benzene or carbon disulphide, and completely soluble in these liquids at a temperature of 25° to 30° C. (77° to 86° F.).

If 1 Gm. of Yellow Wax be boiled for half an hour with 35 Cc. of an aqueous solution of sodium hydroxide (1 in 7), the volume being preserved by the occasional addition of water, the Wax should separate on cooling without rendering the liquid opaque, and no precipitate should be produced in the liquid, after filtration through glass-wool or asbestos, on the addition of hydrochloric acid (absence of *fats* or *fatty acids*, *Japan wax*, or *rosin*). Hydrochloric acid should produce no precipitate in water which has been boiled with a portion of the Wax (absence of *soap*).

If 5 Gm. of Yellow Wax be heated in a flask to 160° C. (320° F.) for fifteen minutes, with 25 Cc. of sulphuric acid, and the mixture then poured into a large excess of water, no notable amount of solid substance, which cannot be decomposed by sulphuric acid on further treatment, should separate (absence of *paraffin* or *ceresin*).

Tests for Identity.—Given above.

Impurities.—Fats, fatty acids, Japan wax, rosin, soap, paraffin, cerasin. Details above.

Saponification Number.—90 to 96. Details in Part V.

The official beeswax consists chiefly of melissyl palmitate, $C_{30}H_{61}-C_{16}H_{31}O_2$, melissyl melissate, $C_{30}H_{61}-C_{29}H_{59}O_2$, and free cerotinic acid.

Yellow wax is defined as a concrete substance prepared by *Apis mellifera*, and is a well-known commercial product, consisting of the comb the bee prepared in which to store its honey, and is supposed to be an excretion of the animal itself.

In obtaining wax the comb is taken, drained of its honey, washed in cold water, and then melted and strained, the product being the bright yellow solid, of pleasant, aromatic odor, having specific gravity 0.955 to 0.967, melting at 63° to 64° C.

The large demand for wax has led to many adulterations of same, the favorite sophistication being rosin or paraffin. For detecting these, neither the specific gravity nor the melting-point is a criterion of quality, for although paraffin has a lower specific gravity than that of wax, and rosin a higher specific gravity, a combination of the two can be made to possess the same specific gravity as pure wax; hence chemical tests of the pharmacopœia are essential for intelligent examination. The test for rosin is that when a sample is boiled with

15 per cent. solution of potassa and filtered, such filtrate should give no precipitation on addition of acid.

In other words, wax itself is unaffected by 15 per cent. potassium hydroxide, whereas the rosin dissolves, forming potassium abietinate. Therefore, if rosin is present, the filtrate contains this potassium abietinate, and the solution when treated with acid precipitates the free abietinic acid.

If the alkaline solution is filtered through paper it will dissolve some of the paper pulp, which will be precipitated on addition of acid, leading to wrong conclusions; hence the present pharmacopœia directs filtration through asbestos.

The test for paraffin is based on the fact that wax is decomposed by boiling with sulphuric acid, while paraffin remains unchanged, as explained under paraffin.

CERA ALBA. White Wax.

Yellow Wax, bleached.

A yellowish-white solid, somewhat translucent in thin layers, having a faint, characteristic odor, and nearly tasteless.

Specific gravity: 0.950 to 0.960 at 25° C. (77° F.).

Melting-point: 64° to 65° C. (147.2° to 149° F.).

In other respects White Wax has the characteristics of, and should respond to, the reactions and tests given under *Cera Flava*.

CETACEUM. Spermaceti.

A peculiar, concrete, fatty substance, obtained from the head of the sperm whale, *Physeter macrocephalus* Linné.

White, somewhat translucent, slightly unctuous masses of a scaly, crystalline fracture and a pearly lustre, with a very faint odor and a bland, mild taste. It becomes yellowish and rancid on prolonged exposure to air.

Specific gravity: 0.938 to 0.944 at 25° C. (77° F.), 0.842 at 100° C. (212° F.).

Melting-point: 45° to 50° C. (113° to 122° F.).

Insoluble in water and nearly so in cold alcohol; soluble in about 50 parts of boiling alcohol; also in ether, chloroform, carbon disulphide, fixed and volatile oils; only slightly soluble in cold petroleum benzin.

An alcoholic solution of Spermaceti is neutral to litmus-paper.

If 1 Gm. of Spermaceti be boiled with 1 Gm. of anhydrous sodium carbonate and 50 Cc. of alcohol, and the mixture cooled and filtered, the filtrate, upon being supersaturated with acetic acid, may become turbid, but it should not afford a precipitate (absence of *stearic acid*).

Tests for Identity.—Given above.

Impurity.—Stearic acid. Details above.

Remarks.—Spermaceti is obtained from the whale by removing from the recently killed animal the oily liquid contained in a cavity in the head. This oil soon congeals when it is submitted to strong pressure. After the liquid part has been expressed, the press cake, crude spermaceti, is purified by melting in water, then boiling with weak alkali, and then washing with water.

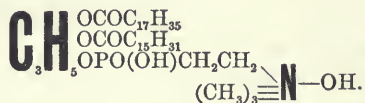
Spermaceti consists chiefly of cetyl palmitate, $C_{16}H_{33}C_{16}H_{33}O_2$ (p. 720), and is a white, somewhat translucent solid, breaking with a scaly fracture, having a specific gravity of 0.945, melting at 50° C. It is a constituent of cold cream, and its chief use is for hardening ointments.

Chinese wax is chiefly ceryl cerotate, $C_{27}H_{55}C_{27}H_{53}O_2$, and is the

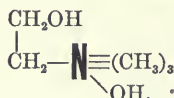
secretion from an insect living on the tree *Ligustrum lucidum*. It is frequently confounded with *Japanese wax*, which is of vegetable origin, being found in the fruit of *Rhus succedanea*.

LECITHIN AND CHOLESTERIN

These two substances can best be discussed at this place, since they are allies of fats. *Lecithin* is a complex ester of glycerin having the graphic formula



On analyzing this formula, we find that the glyceryl group is combined with a stearic acid group, a palmitic acid group, and a phosphoric acid group; while to the latter is attached a choline group,



Cholesterin is an alcohol having the formula $\text{C}_{27}\text{H}_{45}\text{OH}$, and, as mentioned on p. 734, is the alcohol constituent of the ester, wool-fat. Lecithin, choline, and cholesterin are found in animal tissue, such as the brain, yolk of egg, and oxgall (p. 941).

CHAPTER XLII

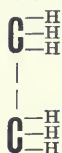
ORGANIC PRODUCTS OF INTERMEDIATE CHARACTER

AMONG the organic chemicals used in pharmacy are several which can scarcely be grouped either as derivatives of hydrocarbons of the fatty series or of the aromatic series. Among these bodies are classed the cyanogen bodies, carbonic acid derivatives, and a few ringed bodies of character midway between the aliphatic compounds already described and the aromatic bodies.

CYANOGEN BODIES

The cyanides can be considered as ethyl or methyl derivatives in which some of the hydrogen is replaced by nitrogen, as can best be shown by comparison of the formulas of ethane and cyanogen:

Ethane.



Cyanogen.



Cyanogen, $(\text{CN})_2$, was discovered by Gay Lussac in 1815, and is made by heating silver cyanide. It is a gaseous body of such poisonous characteristics as to render its manufacture and use unsafe.

A derivative of same, however, paracyanogen, $(\text{C}_2\text{N}_2)_3$, is of interest in pharmacy, as the black precipitate which forms in hydrocyanic acid solution on standing.

Hydrocyanic Acid (HCN) .—This substance was isolated by Scheele in 1782 from oil of bitter almond, and can be considered as methane, in which three of the hydrogen atoms are replaced by one of nitrogen, as shown by comparison of formulas given below:

Methane.



Hydrocyanic acid.



As mentioned above, it is one of the products found in volatile oil of almond, and a description of the process whereby it is extracted will be left until this official is reached. (See p. 778.)

It is prepared by either of the processes given under diluted hydrocyanic acid, or by treating chloroform with ammonia gas under pressure, viz.,



Pure hydrocyanic acid is a very poisonous gas, soluble in water, and used in medicine only in dilute form.

ACIDUM HYDROCYANICUM DILUTUM. Diluted Hydrocyanic Acid.

A liquid composed of not less than 2 per cent., by weight, of absolute Hydrocyanic Acid $[\text{HCN}=26.84]$ and about 98 per cent. of water. It should be kept in small, dark amber-colored, cork-stoppered vials in a cool place. It may be prepared extemporaneously by the following process:

<i>Recipe.</i> —Silver Cyanide, six grammes	6.00 Gm.
Diluted Hydrochloric Acid, fifteen and fifty-four hundredths cubic centimeters	15.54 Cc.
Distilled Water, forty-four and one-tenth cubic centimeters	44.10 Cc.

Mix the Diluted Hydrochloric Acid with the Distilled Water, add the Silver Cyanide, and shake the whole together in a glass-stoppered bottle. When the precipitate has subsided, pour off the clear liquid.

A colorless liquid, of a characteristic odor resembling that of bitter almonds.

On account of its poisonous character it should be tasted with great caution.

It is completely volatilized by heat.

Diluted Hydrocyanic Acid shows an acid reaction with blue litmus-paper.

If to 1 Cc. of the Acid, rendered alkaline by potassium hydroxide T.S., a few drops of ferrous sulphate T.S. be added, and the mixture boiled and then acidulated with hydrochloric acid, a blue precipitate will be formed.

Tests for Identity.—Given above.

Assay.—Details in Part V.

Diluted hydrocyanic acid is an aqueous solution containing 2 per cent. of hydrocyanic acid gas. It is prepared by distilling a mixture

of potassium ferrocyanide with sulphuric acid and water, in a retort, and diluting distillate until it is 2 per cent. strength.

For a simple extemporaneous method of preparing the diluted acid, however, the pharmacopœia directs the treating of silver cyanide with hydrochloric acid and a sufficient quantity of distilled water, the advantage of this process being that it does not require distilling, the silver cyanide combining with the hydrochloric acid and forming insoluble silver chloride, while the hydrocyanic acid goes into solution. In the pharmacopœial recipe a sufficient quantity of water is directed to insure the finished product being exactly 2 per cent.

Dilute hydrocyanic acid is a colorless liquid, which is not very stable, and should be dispensed only when comparatively fresh; hence it is highly advantageous to follow the pharmacopœial alternative process, and manufacture same from silver cyanide.

It possesses a pungent odor (characteristic of bitter almond oil or peach kernel), and should be both smelled and tasted with utmost caution, even in the dilute form. It is used as a sedative and expectorant.

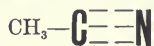
Dose.—0.1 Cc. ($1\frac{1}{2}$ minims).

Mention should be made of the fact that formerly *Scheele's hydrocyanic acid* was a popular form for administering this chemical. It contained 5 per cent. absolute hydrocyanic acid, and the confusion arising from the use of these two strengths of the acid (5 per cent. and 2 per cent.) was positively dangerous. At present, however, Scheele's acid is very rarely employed.

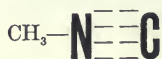
Hydrocyanic acid forms salts with metals, such products being called cyanides. Two of the cyanides are official,—those of potassium and silver,—and each is considered under its respective metal.

Hydrocyanic acid also forms ester-like compounds with organic radicles. As an example might be cited methyl cyanide, CH_3CN . The term cyanide, however, is not applied to these bodies, they being called *nitriles*, and in these bodies the radicle is directly attached to the carbon atom and not to the nitrogen. In this way they differ from the *isonitriles*, which are isomeric bodies in which the radicle is attached to the nitrogen instead of the carbon. The difference between these bodies is best shown by the appended graphic formulas:

Methyl nitrile.



Methyl isonitrile.



Isonitriles are of interest only as being exceedingly poisonous gases of disgusting odor, such as phenyl isonitrile, which is formed in a test for chloroform. (See p. 642.)

Cyanogen forms compounds with iron, forming ferrocyanides and ferricyanides, according to the valence of the metal. One of these (potassium ferrocyanide) is official, while potassium ferricyanide is recognized by the pharmacopœia as a test solution. The former of these bodies has been discussed among the potassium compounds, but this is the proper place for chemical comparison of these two interesting substances.

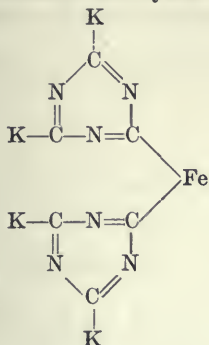
Potassium ferrocyanide, $\text{K}_4\text{Fe}(\text{CN})_6$, may be considered, for the sake of remembering the formula, as a combination of one molecule of ferrous cyanide, $\text{Fe}(\text{CN})_2$, with four molecules of potassium cyanide; that is, $\text{Fe}(\text{CN})_2 + 4\text{KCN}$.

Potassium ferricyanide can be considered, on the other hand, as a combination of ferric cyanide with three molecules of potassium cyanide, that is, $\text{Fe}(\text{CN})_3 + 3\text{KCN}$.

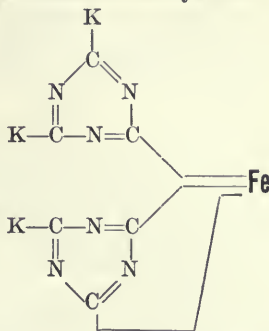
As in the case of the dissection of the alum formula (p. 536), this must be considered as meaning that these two bodies, the ferrocyanide and the ferricyanide, are

mere mechanical combinations of cyanides of potassium and iron, for in both cases there is an intimate combination between the cyanogen group, the iron, and the potassium, as is best shown in the appended graphic formulas:

Potassium ferrocyanide.



Potassium ferricyanide.



As already mentioned on p. 545, the chief pharmaceutic value of these two bodies are as tests for the iron compounds, the ferrocyanide giving blue precipitate with ferric salts and the ferricyanide giving blue only with ferrous salts.

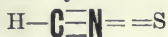
Cyanic Acid (HCNO).—This differs from hydrocyanic acid in containing one atom of oxygen. It in itself possesses no pharmaceutic interest. Its ammonia salt, however, ammonium cyanate, NH_4CNO , is of interest as the product from which urea was first made synthetically (p. 626).

Thiocyanic acid, or *sulphocyanic acid*, HCNS , yields the potassium thiocyanate, KSCN , which is recognized in the pharmacopœia in the form of a test solution, it being the most delicate test for iron, a drop of the solution giving a clear red tint to a liquid containing the merest trace of iron.

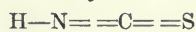
Mercuric thiocyanate, $\text{Hg}(\text{CNS})_2$, is the chief constituent of the dangerous fireworks, "Pharaoh's serpents," these being a mixture of mercury salt with starch or other excipient, and when ignited it swells to enormous proportions, forming shapes somewhat similar to serpents. One of the products of the burning, however, is hydrocyanic acid, and their use by children should be prohibited.

As the organic cyanides possess isomeres which were called isonitriles, so thiocyanic acid possesses an isomere, isothiocyanic acid, the two differing in exactly the same way as the two nitriles, as shown in graphic formulas given below:

Thiocyanic acid.



Isothiocyanic acid.



The allyl ester of isothiocyanic acid, $\text{C}_3\text{H}_5\text{NCS}$, is interesting as the chief constituent of oil of mustard.

OLEUM SINAPIS VOLATILE. Volatile Oil of Mustard.

A volatile oil obtained from Black Mustard (freed from its fatty oil) by maceration with water and subsequent distillation, yielding, when assayed by the process given on p. 1016, not less than 92 per cent. of allyl-isothiocyanate. It should be carefully kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow, limpid, and strongly refractive liquid, having a very pungent and acrid odor. *Great caution should be exercised when smelling this Oil; it should not be tasted without being highly diluted.*

Specific gravity: 1.013 to 1.020 at 25°C . (77°F .).

Miscible with alcohol in all proportions, forming a clear solution.

If to 3 Gm. of the Oil 6 Gm. of sulphuric acid be gradually added, the liquid being kept cool, the mixture, upon subsequent agitation, will evolve sulphur dioxide, but it will remain of a light yellow color, and although at first clear, it will afterward

become thick and occasionally crystalline, and the pungent odor of the Oil will disappear.

If a portion of the Oil be heated in a flask connected with a well-cooled condenser, it should distil completely between 148° and 152° C. (298.4° and 305.6° F.), and both the first and the last portions of the distillate should have the same specific gravity as the original Oil (absence of *alcohol*, *chloroform*, *petroleum*, *fatty oils*, or more than traces of *carbon disulphide*).

If a small portion of the Oil be diluted with 5 times its volume of alcohol, and a drop of ferric chloride T.S. be added, no blue or violet color should be produced (absence of *phenols*).

Tests for Identity.—Given above.

Impurities.—Alcohol, chloroform, petroleum, fatty oil, carbon disulphide, phenols. Details given above.

Assay.—Details in Part V.

Composition.—Chiefly allyl-isothiocyanate and some allyl-cyanide and carbon disulphide.

Remarks.—The manufacture of this volatile oil given in the above official definition will be explained below, when considering constituents of *black mustard*. Oil of mustard is one of the most violently irritating of volatile oils, even pouring of the oil from one bottle to another producing serious irritation to the mucous membrane of the eye and nose, hence the pharmacopœial caution as to smelling.

The assay, based as it is on the behavior of volumetric solution of silver nitrate, will be explained along with that testing liquid (p. 1016).

Medical Properties.—A powerful irritant, used almost exclusively externally as a rubefacient.

Dose.— $\frac{1}{125}$ Cc. ($\frac{1}{8}$ minim).

SINAPIS NIGRA. Black Mustard.

The seed of *Brassica nigra* (Linné) Koch (Fam. *Cruciferae*).

Constituents.—Fixed oil, 25 per cent., the glucoside, *sinigrin*, or *potassium myronate*; the ferment, *myrosin*; mucilage, proteids, etc.

Remarks.—Under glucosides (p. 710) the equation showing the hydrolysis of the glucoside, potassium myronate, into glucose, potassium bisulphate, and allyl-isothiocyanate is given. Here let it be added that the latter substance constitutes the bulk of the volatile oil of mustard. This oil is not found in the dry seed, but is produced when the dry powder is moistened with water by the reaction between the glucoside, potassium myronate, and the ferment, myrosin, both substances occurring in the seed; that black mustard yields its fixed oil on expression, that after the fixed oil is pressed out, the dry squeezed mass, on moistening with water, yields volatile oil by the induced action of ferment on glucoside; and, finally, that the official volatile oil of mustard is obtained by distillation of the moist pulp.

Turning to mustard paper (p. 357), it will be found that in preparing "mustard leaves," as they are usually called, the ground mustard is first percolated with benzin. This solvent extracts the fixed oil, which, if allowed to remain, would grease the paper on which the mustard is spread. Mustard plasters are prepared extemporaneously by mixing powdered mustard with water and spreading on a cloth. The water used in moistening this or the ready-made mus-

tard leaves must be cold, not hot, as hot water destroys the activity of the ferment, myrosin, preventing the formation of volatile oil.

Medical Properties.—Condiment, emetic, rubefacient. Official preparation, *mustard paper* (p. 357).

Dose.—As an emetic, 8 Gm. (120 grains).

SINAPIS ALBA. White Mustard.

The seed of *Sinapis alba* Linné (Fam. *Cruciferae*).

Constituents.—Fixed oil, 25 per cent. The glucoside, *sinalbin*, $C_{30}H_{44}N_2S_2O_{16}$, which hydrolyzes under influence of the ferment myrosin into dextrose, *acrinyl sulphocyanate*, and *sinapin bisulphate* (the last two giving white mustard its activity), proteids, mucilage, *sinapin sulphocyanide*, etc.

Remarks.—It will be seen from the above that the activity of white mustard, like that of black mustard, depends on a sulphurated volatile oil, and likewise is produced by the hydrolysis of a glucoside. It is important to note, however, that neither the glucoside nor the volatile oil has the same composition as those found in black mustard. Both black and white mustard contain some starch, but not as much as do the starch materials with which powdered mustard is sometimes adulterated. To differentiate between natural and excessive amounts of starch, the pharmacopœia provides for both white and black mustard a "limit of starch test" similar to that given for flaxseed (p. 736).

The powdered English mustard used as a condiment possesses a bright yellow tint, while the official mustards, when powdered, are from gray to green-brown. The bright hue of the English article is due to admixture with turmeric, an addition that can scarcely be considered in this case as an adulteration.

Medical Properties and Dose.—Same as black mustard.

Carbonic Acid Derivatives.—As hydrocyanic acid is considered as a methane in which three of the hydrogens are replaced by one of nitrogen, so can carbonic acid be considered as methane, in which two of the hydrogens are replaced by two hydroxyls and two by one atom of oxygen, while formic acid can be considered as methane in which one of the hydrogen atoms is replaced by hydroxyl, and two by one atom of oxygen, as can best be shown by the graphic formulas given below:

Methane.



Carbonic acid.



Formic acid.



Taking the graphic formula of carbonic acid given above as a type, we find several organic bodies which can be considered as direct derivatives thereof. For example, by replacing two hydroxyl groups by chlorine we get a body, COCl_2 , which is called *carbon oxychloride*, or more generally, *phosgene*. This body is made by oxidation of chloroform with chromic acid, and occasionally chloroform decomposes with its formation (particularly under the action of artificial light), in which case the chloroform is, of course, dangerous for inhalation, as explained on p. 642. The hydroxyl groups of carbonic acid can be replaced by other bodies—for example, the amido group, NH_2 . Thus, carbonic acid in which one of the hydroxyl groups is replaced by an amido group gives us carbamic acid, while if the two hydroxyl

groups are replaced by two amido groups, we obtain urea. This can best be explained by examination of the graphic formulas given below:

Carbonic acid.



Carbamic acid.

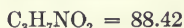


Urea.



Carbamic acid is of interest as being a constituent of the official ammonium carbonate, as explained on p. 489.

ÆTHYLIS CARBAMAS. Ethyl Carbamate.



An ester of carbamic acid [$\text{CO}(\text{OC}_2\text{H}_5)\text{NH}_2$], obtained by the reaction of ethyl alcohol upon urea (carbamide) or one of its salts. It should be kept in well-stoppered bottles.

Colorless, columnar crystals or scales, odorless, and having a cooling, saline taste.

Soluble in less than 1 part of water, 0.6 part of alcohol, 1 part of ether, 1.3 parts of chloroform, and 3 parts of glycerin, at 25° C. (77° F.).

When heated between 47.5° and 50° C. (117.5° and 122° F.) it melts, and at a higher temperature is decomposed, burning without leaving a weighable residue.

If 1 Gm. of Ethyl Carbamate be added to 5 Gm. of sulphuric acid and gently heated, it is decomposed with the evolution of carbon dioxide, while alcohol and acid ammonium sulphate remain in solution.

If 1 Gm. of Ethyl Carbamate be heated with 5 Cc. of concentrated potassium hydroxide solution, ammonia gas is given off, recognizable by the usual tests.

If 0.5 Gm. of Ethyl Carbamate be dissolved in 5 Cc. of water with 1 Gm. of dry sodium carbonate and 0.01 Gm. of iodine, and the solution warmed, yellow crystals of iodoform should separate on cooling.

If 6 Gm. of Ethyl Carbamate be dissolved in 6 Cc. of water, and the solution be divided into 3 equal parts, the addition severally of 5 Cc. of nitric acid, of mercuric nitrate T.S., or of oxalic acid T.S., should not produce a white precipitate (distinction from, and absence of, *urea* or *carbamide*).

Tests for Identity.—Given above.

Impurity.—Urea. Details above.

Remarks.—This body, commonly called *urethane*, is the ethyl ester of carbamic acid, the formula being $\text{CO} < \begin{array}{c} \text{NH}_2 \\ \text{OC}_2\text{H}_5 \end{array}$. It was discovered by Dumas in 1833, and is made by treating phosgene with alcohol and converting the resulting body, the ethyl ester of chlorocarbonic acid, $\text{CO} < \begin{array}{c} \text{Cl} \\ \text{OC}_2\text{H}_5 \end{array}$, into urethane by treatment with ammonia gas.

It is also prepared by treating cyanic acid with alcohol:



It occurs in colorless crystals or scales, and is used as a hypnotic.
Dose.—1 Gm. (15 grains).

Euphorin is phenyl urethane, shown by formula $\text{CO} < \begin{array}{c} \text{NHC}_6\text{H}_5 \\ \text{OC}_2\text{H}_5 \end{array}$.

It will be seen above that urethane is made by treatment of the ethyl ester of chlorocarbonic acid with ammonia gas:



If, instead of ammonia, aniline is used, euphorin results:

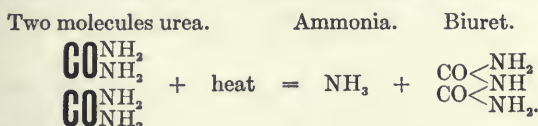


Euphorin is used as an antithermic, antirheumatic, and antiseptic.

Ural is chloral urethane and is hypnotic, as is also *somnal*, ethylated ural, this being made by treating chloral with alcohol and urethane.

Urea, $\text{CO}(\text{NH}_2)_2$.—This body is a normal constituent of the urine, and was found therein in 1773 by Rouelle. Its chief interest is based on the fact that its manufacture was the first organic synthesis performed, this historic work being performed by Wöhler in 1828 by heating ammonium cyanate. It has no interest in pharmacy other than as a constituent of the urine.

By heating urea, *biuret* is obtained by the following reaction:



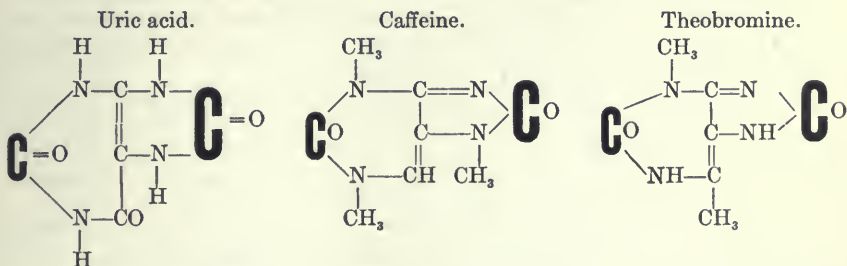
Biuret, when treated with a small quantity of copper sulphate and potassa, gives the characteristic violet-red color, hence the so-called biuret reaction of urea produced on heating urine with these chemicals.

Uric acid was discovered by Scheele in 1776, and is found as a constituent of the urine and of the excrement of serpents. It is made synthetically, but the synthesis is of interest only as establishing its graphic formula, which is given below.

It is generally prepared from *guano*, the dried dung of sea-birds.

Uric acid is interesting to us because of the similarity of its formula to that of the well-known so-called alkaloids, theobromine and caffeine, as is shown by the comparison of the three formulas placed side by side below.

Caffeine is uric acid in which three hydrogen atoms are replaced by three methyl (CH_3) groups, and with the central CO group changed to CH; while theobromine contains only two methyl groups instead of three, as does caffeine.



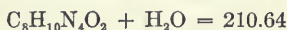
Theobromine is the active constituent of *Theobroma Cacao*, and is obtained from the press cake of the cacao seed (after expressing the oil of theobroma) by mixing it with half its weight of slaked lime and boiling with 80 per cent. alcohol. The crude theobromine obtained from the alcoholic solution is purified by recrystallization.

Theobromine sublimes at 290°C . without melting, and is so sparingly soluble in water that it has to be dispensed as the more soluble alkaline combinations, such

as sodium theobromine. As these alkaline bodies are more or less irritating, double salts, such as diuretin, are to be preferred.

Diuretin is sodium theobromine salicylate, and is made by combining one molecule of sodium theobromine with one molecule of sodium salicylate. It therefore contains 49.7 per cent. theobromine. It occurs in a white powder, which is comparatively unstable in air, breaking up through the loss of carbon dioxide, and for this reason diuretin should never be dispensed on powder papers. It is given as a diuretic in 1-Gm. doses four times a day.

CAFFEINA. Caffeine.



A feebly basic substance [$\text{C}_8\text{H}(\text{CH}_3)_3\text{N}_4\text{O}_2 + \text{H}_2\text{O}$], obtained from the dried leaves of *Thea sinensis* Linné (Fam. *Ternstræmiaceæ*), or from the dried seeds of *Coffea arabica* Linné (Fam. *Rubiaceæ*); found also in other plants.

White, flexible, silky, glistening needles, usually matted together in fleecy masses, permanent in the air; odorless and having a bitter taste. If crystallized from water, it contains one molecule of water of crystallization, but if crystallized from alcohol, chloroform, or ether, it contains none.

Soluble in 45.6 parts of water, 53.2 parts of alcohol, 375 parts of ether, and 8 parts of chloroform at 25° C. (77° F.); soluble in 5.2 parts of water at 80° C. (176° F.) and in 17.1 parts of alcohol at 60° C. (140° F.). Its solubility in water is increased by the presence of certain salts—*e. g.*, potassium bromide, sodium benzoate, sodium salicylate, and others.

Caffeine sublimates at about 178° C. (352.4° F.) and should leave no residue. Its melting-point is 236.8° C. (458.3° F.).

An aqueous solution of Caffeine is neutral to litmus-paper.

If a small quantity of Caffeine be dissolved in about 1 Cc. of hydrochloric acid in a porcelain dish, a little potassium chlorate added, the whole evaporated to dryness on a water-bath, and the dish then inverted over a vessel containing a few drops of ammonia water, the residue will acquire a rich purple color, which is destroyed by fixed alkalis.

If a fragment of Caffeine be dissolved in sulphuric acid and a minute fragment of potassium dichromate be added to the liquid, a yellowish-green color, which gradually becomes green, will be produced.

Caffeine should dissolve in sulphuric acid or in nitric acid without producing a color (absence of *organic impurities*).

Its aqueous solution should not be precipitated by mercuric potassium iodide T.S. (absence of *alkaloids*).

Tests for Identity.—Given above.

Color Tests.—See above.

Impurities.—Organic impurities, alkaloids. Details above.

Manufacture.—Made chiefly from the dust and sweepings of tea-leaves; treating a decoction of same with lead acetate, precipitating excess of lead with hydrogen sulphide, and finally concentrating solution to point at which it crystallizes.

It can also be made from tea, coffee, or guarana by cooking with litharge and water, precipitating excess of lead with hydrogen sulphide, and crystallizing. As the yield from coffee is small (0.87 to 2.0 per cent.), tea sweepings (1.5 per cent. to 3 per cent.) or guarana (5 per cent.) are generally used.

Caffeine was formerly considered an alkaloid, but is now, as mentioned above, considered as a feebly basic principle. It differs from alkaloids in not being precipitated by potassio-mercuric iodide. Like alkaloids, it forms combinations with acids, but these possess such weak union that the pharmacopœia, in the case of the one official acidulated compound, that with citric acid, does not call it caffeine citrate, but citrated caffeine.

Dose.—65 milligrammes (1 grain).

CAFFEINA CITRATA. Citrated Caffeine.

<i>Recipe.</i> —Caffeine, <i>fifty grammes</i>	50 Gm.
Citric Acid, <i>fifty grammes</i>	50 Gm.
Distilled Water, hot, <i>one hundred cubic centimeters</i>	100 Cc.

Dissolve the Citric Acid in the hot Distilled Water, add the Caffeine, and evaporate the resulting solution, on a water-bath, to dryness, constantly stirring toward the end of the operation. Reduce the product to a fine powder and transfer it to well-stoppered bottles.

A white powder, odorless, having a slightly bitter, acid taste, and an acid reaction.

One part of Citrated Caffeine forms a clear, syrupy solution, with about 4 parts of hot water. Upon dilution with 5 parts of water, a white, crystalline precipitate (caffeine) separates, which redissolves when about 25 parts of water have been added. It is also soluble in a mixture of equal volumes of chloroform and alcohol.

If 0.25 Gm. of Citrated Caffeine be mixed with 5 Cc. of concentrated sulphuric acid in a porcelain dish, the mixture protected from dust and heated for fifteen minutes on a water-bath, a lemon-yellow color, and not a brown or black color, should develop (absence of *tartaric acid*).

Tests for Identity.—Given above.

Impurities.—Tartaric acid. Details above.

Remarks.—Citrated caffeine, as given above, is made by blending caffeine with citric acid. The union is not a definite one, like the alkaloidal salts, hence the product is called “citrated,” and not “citrate of.”

It is chiefly used for making the effervescent salt.

Dose.—125 milligrammes (2 grains).

CAFFEINA CITRATA EFFERVESCENS. Effervescent Citrated Caffeine.

<i>Recipe.</i> —Citrated Caffeine, <i>forty grammes</i>	40 Gm.
Sodium Bicarbonate, dried and powdered, <i>five hundred and seventy grammes</i>	570 Gm.
Tartaric Acid, dried and powdered, <i>three hundred grammes</i>	300 Gm.
Citric Acid, uneffloresced crystals, <i>one hundred and ninety-five grammes</i>	195 Gm.
To make about <i>one thousand grammes</i> ..	1000 Gm.

Powder the Citric Acid and mix it intimately with the Citrated Caffeine and Tartaric Acid, then thoroughly incorporate the Sodium Bicarbonate. Place the mixed powders on a plate of glass or in a suitable dish, in an oven heated to between 93° and 104° C. (199.4° and 219.2° F.).

When the mixture has acquired a moist consistence by the aid of a careful manipulation with a wooden spatula, rub it through a No. 6 tinned-iron sieve, and dry the granules at a temperature not exceeding 54° C. (129.2° F.). Keep the product in well-stoppered bottles.

Remarks.—Effervescent citrated caffeine is made similar to the other effervescent salts, viz., by rubbing the chemical (citrated caffeine in this case) with sodium bicarbonate, tartaric acid, and citric acid, softening mass by water-bath heat, and granulating by running through a coarse sieve. This product is a pleasant effervescent remedy for headache and nervous disorders, and is intended to replace the many proprietary preparations of similar character on the market.

Dose.—4 Gm. (60 grains).

DRUGS CONTAINING CAFFEINE

GUARANA. *Guarana.*

A dried paste consisting chiefly of the crushed seeds of *Paullinia Cupana* Kunth (Fam. *Sapindaceæ*), yielding, when assayed by the process given on p. 1023, not less than 3.5 per cent. of its alkaloidal principles.

Constituents.—Caffeine, usually 5 per cent., paullitannic acid, gum, resin, volatile oil, etc.

Remarks.—Guarana is the seed of *Paullinia Cupana*, ground up, made into a paste with water, rolled into cylinders, and dried, either by the sun or by the heat of a slow fire. Its manufacture is in the hands of the Brazilian Indians, and is most primitive. The active ingredient of guarana is the same as that found in coffee and tea—the official caffeine. The pharmacopœial assay will be explained in Part V.

Medical Properties.—Stimulant, somewhat diuretic. Administered in the form of the official *fluidextract* (p. 247), the elixir is a popular preparation, and the recipe for it will be found in the National Formulary.

Dose.—Of drug, 2 Gm. (30 grains).

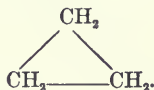
Kola, the seed of *Sterculia acuminata*, has lately come into popularity as a stimulant, owing this property to the caffeine it contains.

Coffee, the roasted seed of *Coffea Arabica*, and tea, the leaves of *Thea chinensis*, also owe their activity to caffeine.

SIMPLER RINGED COMPOUNDS

Up to this time we have considered compounds in which the carbon atoms are linked together, one after another, in the nature of the links of a chain, but even as the end links of a chain can be in turn linked, forming an endless chain or ring, so can end carbon atoms under certain circumstances be persuaded to link together, forming organic bodies of a ring nature. We have already discussed unsaturated chain compound,

Propylene, $\text{CH}_2=\text{CH}-\text{CH}_3$, and from it can be made the isomeric *trimethylene*, having the formula

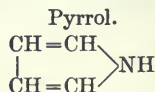
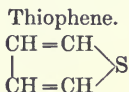
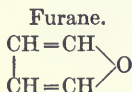


This represents the simplest ring.

In **furane**, $\text{C}_4\text{H}_4\text{O}$, we have a ring of four carbon atoms in which the end carbon atoms, however, are not directly combined, but are linked by aid of an oxygen atom, as shown in graphic formula.

In **thiophene**, $\text{C}_4\text{H}_4\text{S}$, we have a similar body, in which the end carbon atoms are linked by aid of the sulphur atom.

Pyrrol, $\text{C}_4\text{H}_4\text{NH}$, is a similar body in which the end carbon atoms are attached by means of the NH group, these three being expressed below by graphic formulas:



Of these three bodies, furane and thiophene are of little pharmaceutic interest, the latter substance being discovered by Victor Meyer as a constituent of benzene, with which, curious to relate, it has not merely been combined without discovery, but actually gave chemical test, which, for a long period, masqueraded as a test for the identity of benzene.

Pyrrol is of interest to us chiefly as the basis of one of the best known iodoform substitutes, *iodol*.

IODOLUM. Iodol.

$$C_4I_4.NH = 566.17$$

Tetraiodopyrrol, a derivative of the base pyrrol, obtained by the direct action of iodine upon the base in the presence of alcohol. Iodol should be preserved in amber-colored bottles, protected from light.

A light grayish-brown, crystalline powder, without odor or taste.

It is soluble in about 4900 parts of water, 9 parts of alcohol, 1.5 parts of ether, and 105 parts of chloroform at 25° C. (77° F.); soluble in fixed oils. It is soluble in concentrated sulphuric acid, producing a green solution, gradually changing to brown.

When heated to 100° C. (212° F.) it remains unchanged, but at a temperature of 140° to 150° C. (284° to 302° F.) it is decomposed with the liberation of violet iodine vapors. When ignited it should leave not more than 0.1 per cent. of residue (limit of *inorganic impurities*).

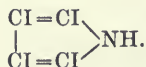
If 0.5 Gm. of Iodol be shaken with 100 Cc. of water and filtered, the filtrate should not be made more than slightly opalescent by silver nitrate T.S. (absence of *hydriodic acid* or soluble metallic *iodides*).

If 0.5 Gm. of Iodol be shaken with 100 Cc. of water and filtered, the filtrate should not communicate more than a light yellowish tinge to chloroform (absence of appreciable amount of *free iodine*).

Tests for Identity —Given above.

Impurities.—Inorganic impurities, hydriodic acid, iodides, more than traces of free iodine. Details above.

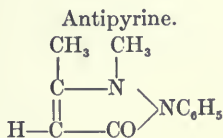
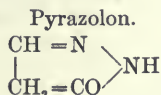
Remarks.—Iodol was discovered by Ciarnian and Silber in 1885, being tetraiodopyrrol, as shown by the following formula:

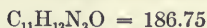


It is made by directly combining iodine with pyrrol, and has the highest iodine contents of any organic substance save iodoform, and is greatly superior to that body in being devoid of its disagreeable odor. It is a useful antiseptic.

Dose.—250 milligrammes (4 grains).

A body of composition similar to pyrrol is *pyrazol*, and a derivative of this, phenyl-dimethyl-pyrazolon, is the valuable synthetic, *anti-pyrine*. Below is given the formula of each of these substances for sake of comparison:



ANTIPYRINA. Antipyrine.

Phenyldimethylpyrazolon $[\text{C}_3\text{HN}_2\text{O}(\text{CH}_3)_2.\text{C}_6\text{H}_5]$, obtained by the condensation of phenylhydrazine with aceto-acetic ether, and methylation of the product.

A colorless, almost odorless, crystalline powder or tabular crystals, with a slightly bitter taste.

Soluble in less than 1 part of water, in 1 part of alcohol, 1 part of chloroform, and in 30 parts of ether at 25°C . (77°F .).

When heated to 113°C . (235.4°F .) it melts. Upon ignition it is consumed without leaving a weighable residue.

The aqueous solution has a neutral reaction upon litmus-paper, but Antipyrine unites directly with acids to form salts.

If to an aqueous solution of Antipyrine, tannic acid T.S. be added, an abundant white precipitate (tannate) is formed.

If 0.1 Gm. of sodium nitrite and 12 Cc. of an aqueous solution of Antipyrine (1 in 100) be mixed, a nearly colorless liquid is obtained, which, upon the addition of 1 Cc. of diluted sulphuric acid, develops a deep green color (formation of *isonitroso-antipyrine*).

If to 2 Cc. of a dilute aqueous solution of Antipyrine (1 in 1000) 1 drop of ferric chloride T.S. be added, a deep red color is produced, which upon the addition of 10 drops of sulphuric acid is changed to light yellow.

Two Cc. of an aqueous solution of Antipyrine (1 in 100) mixed with an equal volume of nitric acid assumes a yellowish color, passing to crimson on warming (distinction from *acetanilide* and *acetphenetidin*).

On warming 0.1 Gm. of Antipyrine with sodium hydroxide T.S., and again warming after the addition of chloroform, the disagreeable odor of phenyl-isocyanide should not be developed (absence of *acetanilide*).

Tests for Identity.—Given above.

Impurities.—Acetanilid, acetphenetidin. Details given above.

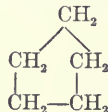
Remarks.—Antipyrine is made by treating ethyl-diacetic-ester with phenylmethyl-hydrazine. It occurs in the form of a white powder, soluble in one part of water, and melting at about 113°C . Its most likely substitute, antifebrin, melts at about the same point, but it is interesting to note that antipyrine mixed with antifebrin does not melt at 113°C ., but at 45°C ., so the melting-point is a valuable means of detecting admixture.

Antipyrine is one of the most valuable of the antipyretics, the consumption in America and Europe being enormous. It was not recognized by the pharmacopœia until this edition, as it was a monopoly, the patent being controlled by a German chemical firm. Its use is not free from danger, hence pharmacists should be careful in recommending it. In fact, it should not be administered except by direction of a physician.

Dose.—250 milligrammes (4 grains).

The large demand for antipyrine gave rise to the manufacture of chemicals of similar character. Among these are *benzopyrine* (antipyrine benzoate), *salipyrine* (antipyrine salicylate), and *iodopyrine*, with an atom of iodine replacing an atom of hydrogen in phenyl compound. *Hypnal* is a combination of antipyrine and chloral.

We have just mentioned ring compounds containing three and four carbon atoms respectively; a similar ring body, containing five carbon atoms, is pentamethylene:

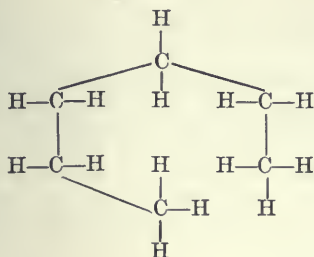


This is of no interest in pharmacy, and is merely mentioned as a step toward the ring compounds of real value—those possessing six carbon atoms.

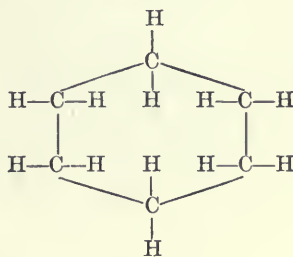
The saturated ring compound having six carbon atoms has the formula C_6H_{12} .

This body can be considered as hexane (C_6H_{14}) (Formula I), with a hydrogen atom from each end carbon atom, which are then linked by the free bonds, giving a formula as shown in Formula II.

FORMULA I.



FORMULA II.



This body, C_6H_{12} , is called hexamethylene or hexahydrobenzene.

HEXAMETHYLENAMINA. Hexamethylenamine.

$$C_6H_{12}N_4 = 139.18$$

A condensation product [Hexamethylene-tetramine, $(CH_2)_6N_4$], obtained by the action of ammonia upon formaldehyde. It should be kept in well-stoppered bottles.

Colorless, lustrous, odorless crystals, having, when in aqueous solution, an alkaline reaction upon red litmus-paper.

Soluble in about 1.5 parts of water, 10 parts of alcohol, and in 228 parts of ether at $25^\circ C.$ ($77^\circ F.$); in 1.5 parts of water at $100^\circ C.$ ($212^\circ F.$) and in about 8 parts of hot alcohol.

When heated to $263^\circ C.$ ($505.4^\circ F.$) it sublimes without melting and with partial decomposition.

If 0.1 Gm. of Hexamethylenamine be mixed with 0.1 Gm. of salicylic acid and 5 Cc. of sulphuric acid, and then heated moderately, a carmine-red color should be produced.

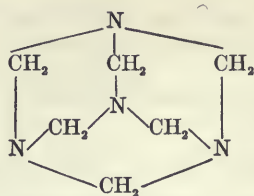
If an aqueous solution of Hexamethylenamine (1 in 10) be heated with diluted sulphuric acid, it is decomposed with the liberation of formaldehyde (recognized by its odor or by its darkening paper moistened with silver ammonium nitrate T.S.).

If an aqueous solution (1 in 10) be heated with diluted sulphuric acid and then supersaturated with solution of sodium hydroxide, ammonia is liberated.

If to an aqueous solution (1 in 10) tannic acid T.S. be added, a precipitate is formed. If mercuric chloride T.S. be added to an aqueous solution (1 in 10), a precipitate is produced which on standing forms crystalline needles.

Tests for Identity.—Given above.

Remarks.—While hexamethylenetetramine can in some respects be best discussed at this place, from the purely chemical standpoint it might be better considered as a condensation product of formaldehyde, from which it is made. In truth, it has but little in common with hexamethylene, discussed above, its structure being quite different from that chemical, its graphic formula being—



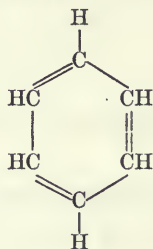
It is known in commerce as *cystogen*, *formin*, *urotropin*, etc., and is used as a uric-acid solvent and urinary antiseptic.

Dose.—250 milligrammes (4 grains).

From hexamethylene (C₆H₁₂) it is but a simple step (theoretically) to benzene, C₆H₆ (or benzol).

If from the graphic formula of hexamethylene given above the six hydrogen atoms inside the hexagon are removed, and freed bonds of the carbon atoms are linked, we get the formula of benzene:

FORMULA III.



Observe in the latter formula we have a hexagon or ring of six carbon atoms, each linked to one neighbor by one bond, and to the other by two or by "a double bond," as we say. We have stepped from the fatty to the aromatic hydrocarbons—from the chain to the ring series.

CHAPTER XLIII

THE AROMATIC SERIES

BENZENE AND ITS DERIVATIVES

UP to this page, Part IV. has been devoted to the chain of fatty series, they being so called because a large number of their compounds were of fatty character. The next class of organic chemicals to consider is the aromatic series; so called because, among them, are found most of the aromatic substances in general use. All contain as a nucleus a ring compound, based on the benzene type of hydrocarbons.

The graphic formula of benzene given in the preceding chapter is the one generally accepted, it answering best all the requirements of a body having the formula C₆H₆.

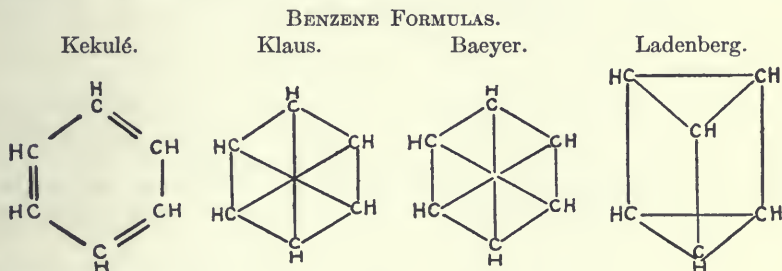
This formula is the one suggested by Professor Kekulé, of Bonn,

whose work on this very difficult subject has given him a place among the great philosophers, and one of the most striking proofs of the truth of Kekulé's formula is the fact that it has withstood the criticism of fifty years, and is more firmly accepted now than ever before.

The chief difficulty of ascribing graphic formulas of the body C_6H_6 was found in reconciling the formula to the tetravalency of the carbon atoms. As shown on the preceding page, there is known a ring compound containing six carbon atoms united by single bonds. This means two hydrogen atoms attached to each carbon atom, a body having the formula C_6H_{12} . For a body to have the formula C_6H_6 means that every one of the six carbon atoms will have one unsaturated bond, a theory first advanced by Kekulé.

Other graphic formulas for benzene have been suggested, and these are pictured below. Thus, in Klaus' formula, the extra bonds of the carbon atoms are placed diagonally from carbon to carbon atom.

Baeyer considers these extra bonds as gravitating toward the center of the compound without forming a distinct union with any other bond, while Ladenberg assumes a totally different form, pictured below and called the prism form:



Each of these separate formulas has its advantages, and each shows some difficulties in reconciling them to the facts. And, after all the arguments are weighed, the time-honored Kekulé formula seems the most satisfactory.

Benzene is what is commercially called benzol, preference being given to the first name, because of the desire to limit the termination "ol" to the alcohols. In this case the change is unfortunate, inasmuch as it leads to confusion between this aromatic body and the official benzin, which is a petroleum product, of character totally different from the benzene of which we are now speaking.

Benzene was discovered by Faraday in 1825. It was found in coal-tar in 1845 by Hoffman, whose researches into the formation of dye-stuffs was a wonderful impetus to the benzene industry. Benzene can be made by distilling benzoic acid with chalk, but now practically the entire output is from coal-tar, that side-product in the manufacture of coal-gas from which so many valuable synthetics have been made in recent years.

Benzene in itself has comparatively limited use, being employed almost entirely as a solvent for fats and the like. Its derivatives, however, are very numerous, and many of them of great commercial value.

The statement given above of the danger of confusion of benzene with benzin makes a simple test of distinction between these two bodies advisable, and this we have in their action on carbolic acid. Benzin does not dissolve phenol, while benzene does. By treating benzene with concentrated nitric acid and subsequent distillation we are able to replace one of the hydrogens of benzene by an NO_2 group, giving us nitrobenzene, $\text{C}_6\text{H}_5\text{NO}_2$, which is commercially called *oil of mirbane*, and very erroneously called *artificial oil of bitter almond*, by reason of its somewhat similar odor. Oil of mirbane is used as a perfume for cheap soap.

Phenols are a class of alcohols peculiar to the benzene series, the hydroxyl being attached directly to the benzene nucleus, and its simplest example is the ordinary phenol, which is generally known as carbolic acid, and which can be considered as the hydroxide of *phenyl*, the latter word being the designation of the radicle, C_6H_5 . These phenyls are like alcohols, inasmuch as they form ethers, an example of which is the phenyl methyl ether, $\text{C}_6\text{H}_5\text{OCH}_3$, commercially known as anisol, and, like the alcohols, form esters, an example of which is the official salol (phenyl salicylate). However they are unlike the primary alcohols, inasmuch as they do not oxidize to aldehydes, and in most reactions will resemble the tertiary alcohols, it being a very difficult matter to oxidize same, although ketones are sometimes formed.

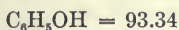
As stated above, the best known phenol is what is known as carbolic acid, and this is called an "acid" because it forms salts with the alkalis. From it, for example, we obtain sodium carbolate ($\text{C}_6\text{H}_5\text{ONa}$) and potassium carbolate ($\text{C}_6\text{H}_5\text{OK}$). It must be recalled, however, that even the alcohols of the chain series yield similar compounds, for instance, sodium alcoholate, $\text{C}_6\text{H}_5\text{ONa}$ (p. 655). The carbolate, however, differs from the alcoholate, inasmuch as this can be made from carbolic acid by simply treating with alkali, whereas to make a sodium compound from alcohol requires treatment with the metal itself.

The phenols possess most of the reactions of the benzene nucleus from which they are derived; that is, the hydrogen of the benzene nucleus can be replaced by chlorine, the NO_2 group, and the like. An official illustration of such a modified phenol obtained is sodium sulphocarbonate (p. 469).

The following derivatives of benzene are of interest in pharmacy:

Phenol.....	$\text{C}_6\text{H}_5\text{OH}$.
Liquefied phenol.....	86 per cent. solution of phenol.
Crude carbolic acid, U. S. P. 1890.	
Anisol.....	$\text{C}_6\text{H}_5\text{—OCH}_3$.
Phenol sulphuric acid.....	$\text{C}_6\text{H}_4\begin{smallmatrix} \text{OH} \\ \text{SO}_3\text{H} \end{smallmatrix}$.
Guaiacol.....	$\text{C}_6\text{H}_4\begin{smallmatrix} \text{OH} \\ \text{OCH}_3 \end{smallmatrix}$.
Guaiacol carbonate.....	$(\text{C}_6\text{H}_4\text{OCH}_3\text{O})_2\text{CO}$.
Resorcinol.....	$\text{C}_6\text{H}_4(\text{OH})_2$.
Pyrogallol.....	$\text{C}_6\text{H}_3(\text{OH})_3$.
Anilin.....	$\text{C}_6\text{H}_5\text{NH}_2$.
Methylthionine hydrochloride.....	$\text{C}_6\text{H}_5\text{N}_3\text{SCL}$.
Acetanilide.....	$\text{C}_6\text{H}_5\text{NHC}_2\text{H}_3\text{O}$.
Acetphenetidine.....	$\text{C}_6\text{H}_4\begin{smallmatrix} \text{OC}_2\text{H}_5 \\ \text{NHC}_2\text{H}_3\text{O} \end{smallmatrix}$.

PHENOL. Phenol.



[ACIDUM CARBOLICUM, PHARM. 1890]

Hydroxybenzene, obtained either from coal-tar by fractional distillation, and subsequent purification, or made synthetically. It should contain, when assayed by the process given below, not less than 96 per cent. of absolute Phenol. It should be kept in dark amber-colored, well-stoppered bottles.

Colorless, interlaced, or separate needle-shaped crystals, or a white, crystalline mass, sometimes acquiring a reddish tint; having a characteristic, somewhat aromatic odor; when copiously diluted with water, it has a sweetish taste, with a slightly burning after-taste, and, when undiluted, cauterizes and whitens the skin and mucous membrane.

Soluble in 19.6 parts of water at 25° C. (77° F.), the solubility varying according to the degree of hydration of the Phenol; very soluble in alcohol, ether, chloroform, benzene, carbon disulphide, glycerin, fixed and volatile oils; almost insoluble in petroleum benzin.

When gently heated, Phenol melts, forming a highly refractive liquid. It is also liquefied by the addition of about 8 per cent. of water. If the Phenol be liquefied by a gentle heat, and then slowly cooled under constant stirring, until it is partly recrystallized, the semiliquid mass should have a temperature (remaining stationary for a short time) not lower than 40° C. (104° F.).

Phenol should have a boiling-point not higher than 188° C. (370.4° F.). A lower boiling-point or a higher melting-point indicates a less hydrated Phenol.

When heated upon a water-bath it should be volatilized without leaving a residue. The vapor is inflammable.

Phenol is faintly acid to blue litmus-paper.

Its aqueous solution yields, with bromine water, a white precipitate of tribromophenol, which at first redissolves, but becomes permanent as more of the reagent is added, and appears crystalline when viewed under the microscope.

On adding to 10 Cc. of an aqueous solution of Phenol (1 in 100) 1 drop of ferric chloride T.S., the liquid acquires a violet-blue color, which is permanent.

One volume of cold, liquefied Phenol (rendered liquid by the addition of 8 per cent. of water) forms, with 1 volume of glycerin, a clear liquid which is not rendered turbid by the addition of 3 volumes of water (absence of *creosote* and of *cresol*).

Tests for Identity.—Given above.

Impurities.—*Creosote* and *cresol*. Details above.

Assay.—Details in Part V.

Remarks.—This phenol was discovered by Runge, in 1834, as a constituent of coal-tar, from which it is separated by contact with potassium hydroxide (thus forming potassium carbolate); separation of the acid from potassium carbolate by treating with sulphuric acid and fractional distillation of the freed product. Carbolic acid has also been made synthetically from benzene by conversion of benzene into benzosulphonic acid, $\text{C}_6\text{H}_5\text{SO}_3\text{Na}$, and fusing this with sodium hydroxide, when sodium carbolate and sodium sulphite are produced:



The official carbolic acid occurs in colorless, needle-shaped crystals having a characteristic odor and sweetish taste. It should, however, never be tasted in concentrated form.

The solubility of the crystalline carbolic acid is a matter of interest. *One hundred parts dissolve 8 parts of water* to a clear liquid. If more water be added, it clouds, since the acid will dissolve no more. This cloudiness is continued until 1500 parts of water have been added, when the conditions are reversed and the water dissolves the acid.

Some of the crystalline carbolic acids, especially those of English

PRINCIPLES OF PHARMACY

re, acquire a reddish tint on standing, and the origin of this disagreeable phenomenon is still a matter of question. Some claim that the nitrogen of the air acts on the phenol, forming one of the numerous dye-stuffs obtained from benzene. Others think it due to organic impurities, but Schneider furnishes strong proof that copper contamination is the cause. The removal of this color is a matter of considerable difficulty; the only perfect way is by the redistillation of the carbolic acid, an operation that is not safe for an ordinary pharmacist.

It must be borne in mind that the vapors of carbolic acid are inflammable, and, therefore, great care should be used in melting same by means of heat; in fact, the best way to melt the substance is to dip the bottle in heated water rather than to heat the mass direct, even on a water-bath.

Carbolic acid has a number of characteristic reactions, among which may be cited the violet-blue color it gives with a drop of ferric chloride solution, its coagulating action on albumin and collodion, and the yellow precipitate with bromine. The first two tests are of interest as distinguishing between itself and creosote, carbolic acid being a frequent substitute for the more costly wood-tar products. The bromine reaction is of interest as the basis of the volumetric estimate of carbolic acid (see Part V).

As mentioned above, carbolic acid can be liquefied by addition of 8 per cent. of water. This solution, however, is scarcely as desirable as one liquefied by an addition of a small quantity of alcohol although even that may be objectionable on account of possible irritating qualities of the alcohol. This solution, the so-called 95 per cent. carbolic acid, is what is generally dispensed in answer to calls for pure carbolic acid instead of the crystals themselves. The extended use of this 95 per cent. carbolic acid by suicides, and the almost invariable fatal results from use in this way, render it necessary for pharmacists to use great caution in selling same. In the city of Cleveland the pharmacists have attempted to introduce for general dispensing purposes a 33 per cent. carbolic acid, made by mixing the crystals with a sufficient quantity of glycerin. This solution serves all the legitimate purposes of carbolic acid, is freely miscible with water in all proportions, and is of sufficient strength for any real needs for this antiseptic; while, on the other hand, if the would-be suicide administers same, corrosive action is scarcely likely to occur, and this is usually the cause of death. Such carbolic acid is sold in Cleveland under the name of "carbolic acid No. 33."

Carbolic acid is one of the most valuable of all antiseptics, its germicidal action being due to the fact that it coagulates albumin. In full strength it is, as mentioned above, a very caustic poison, the antidote for which is sodium sulphate, followed by mucilaginous drinks. Recent investigation shows that oil should never be used for an antidote for carbolic acid, for while oil lessens the caustic effect by dissolving the carbolic acid, it then brings it to a form best adapted to assimilation by the system; in other words, while being an antidote for the caustic effect of the poison, it opens the way for it to exert its physiologic influence.

Sodium sulphate is suggested as an antidote because it forms

with carbolic acid the harmless sodium sulphocarbolate, and this being produced, the escharotic action of the acid is relieved by mucilaginous liquids, such as mucilage of flax-seed. Alcohol has also been suggested as an antidote for carbolic acid. The official preparations of carbolic acid are a 5 per cent. ointment and the glycerite, which contains 20 per cent. of the phenol. In some sections *carbolic oil*, made by combining phenol with either olive oil or cotton seed oil, in proportions from one to eight, is a favorite remedy for cuts and sores.

Dose.—65 milligrammes (1 grain).

PHENOL LIQUEFACTUM. Liquefied Phenol.

A liquid composed of not less than 86.4 per cent., by weight, of absolute Phenol [$C_6H_5OH=93.34$] and about 13.6 per cent., by weight, of water.

Recipe.—Phenol, a convenient quantity.

Distilled Water, a sufficient quantity.

Liquefy the Phenol by placing the unstoppered container in a water-bath, and apply heat gradually until the crystals have melted; transfer the liquid to a tared vessel and weigh; then add for each *nine grammes* of Phenol *one gramme* of Distilled Water and mix thoroughly. It should be kept in dark amber-colored, well-stoppered bottles.

A colorless liquid, which may develop a slight reddish tint upon keeping, having a characteristic, somewhat aromatic odor, and when copiously diluted with water, a sweetish taste with a slightly burning after-taste, and, when undiluted, cauterizing and whitening the skin and mucous membrane.

Specific gravity: about 1.065 at 25° C. (77° F.).

Soluble in 12 parts of water at 25° C. (77° F.). Miscible with alcohol, ether, and glycerin in all proportions.

One part of Liquefied Phenol acquires a permanent cloudiness when mixed respectively with 2 parts of chloroform, 1.5 parts of benzene, 2.5 parts of carbon disulphide, 2 parts of turpentine, or 2.5 parts of olive oil.

Liquefied Phenol begins to crystallize when the temperature of the liquid is lowered to about 13.5° C. (56.3° F.).

When heated, Liquefied Phenol begins to boil at about 115° C. (239° F.), and upon continuing the heat the boiling-point rises; it should not exceed 188° C. (370.4° F.). When thus deprived of water, it should respond to the tests given under Phenol.

Remarks.—As mentioned under Phenol, that chemical forms a permanent liquid by the addition of from 8 to 10 per cent. water, and this liquid has been given pharmacopoeial recognition. Its use is, of course, the same as phenol.

Dose.—0.05 Cc. (1 minim).

Acidum Carbolicum Crudum (U. S. P. 1890).—*Crude carbolic acid* is a liquid consisting of various constituents of coal-tar obtained by fractional distillation. The official crude carbolic acid is a nearly colorless or reddish or brownish-red liquid, and possesses a characteristic odor. The true carbolic acid ordinarily sold in stores represents a very dirty black liquid, which has but little value, as it is notoriously weakened by addition of water. Crude carbolic acid is used entirely as a disinfectant, for which purpose it is better than the crystals, because it contains a large quantity of cresol in addition to its chief constituent, phenol.

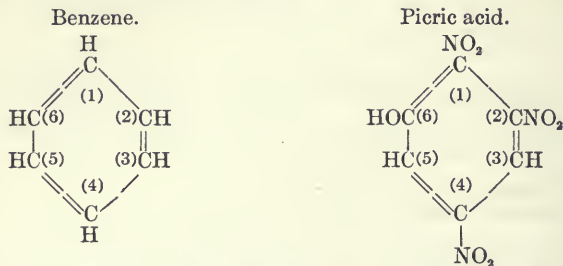
Anisol, $C_6H_5OCH_3$, as mentioned above, is phenylmethyl-ether. We have already learned that if methyl alcohol and ethyl alcohol are combined in the presence of sulphuric acid, there will be yielded methyl-ethyl-ether, and in the same way if methyl alcohol and phenol are mixed in the presence of sulphuric acid, methylphenyl-ether, or anisol, is produced. As its name suggests, it is the chief constituent of oil of anise.

As the consideration of methyl salicylate was deferred until we had discussed

salicylic acid, it is consistent to refer the official *phenyl salicylate*, or *salol*, to the same heading.

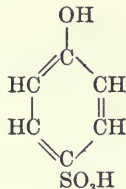
Picric acid, $C_6H_2(NO_2)_3OH$, is a trinitrophenol; that is, it is phenol in which three of the hydrogen atoms of the benzene nucleus are replaced by NO_2 groups, these groups replacing the hydrogen attached to carbon atoms one, two, and four respectively.

At this place it might be stated that, for purposes of convenience, chemists have numbered the carbon atoms of the benzene ring as shown below:



Picric acid is made by treating trinitrobenzene with potassium ferricyanide. It appears in bright yellow crystals, soluble in water, having a strong acid character, and very explosive, hence should be handled by the pharmacist with the utmost care. It is not used much in pharmacy, its chief value being as a dye for silk; in certain countries it is used as an explosive, and in chemistry it is used as a reagent for alkaloids, and also for albumin and sugar in urinalysis.

Sulphocarboic acid (or phenol sulphonic acid) is a body prepared by treating carboic acid with sulphuric acid. Its composition is best understood from the graphic formula:



The official sodium salt of this acid is made by treating mixture of sulphuric acid and carboic acid with barium carbonate, whereby solution of barium sulphocarbonate is produced, and this solution, on being treated with a molecular quantity of sodium carbonate, produces sodium sulphocarbonate, while the barium is precipitated as barium carbonate. (See Sodium Compounds, p. 469.)

Soziodol represents sulphocarboic acid in which two of the hydrogen atoms of the benzene nucleus have been replaced by iodine, and, indeed, those hydrogens attached to the carbon atoms numbers two and six.

This is used as an antiseptic in the form of ammonium, barium, lead, and other metallic salts.

Sozol is a trade name for aluminum sulphocarbonate.

Aseptol is an aqueous solution of orthosulphocarboic acid. This body differs from sulphocarboic acid, mentioned above, in having the hydroxyl and sulphonic acid (SO_3H) groups on adjoining carbon atoms, this distinction between ortho-, meta-, and para-compounds being reserved for discussion later.

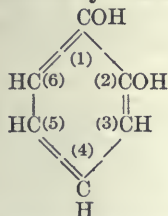
DIATOMIC PHENOLS

As we already learned when considering the fatty series, besides alcohols containing one hydroxyl, similar bodies have been found containing two, three, and even more hydroxyls, these being examples of polyatomic alcohols. Thus, glycerin, which, it will be recalled, has

the formula $C_3H_5(OH)_3$, is a polyatomic alcohol. In the same way we can have polyatomic phenols.

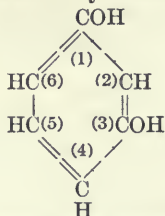
The simplest of these phenols are the diatomic—those possessing two hydroxyl groups. The direct benzene derivatives of this class have the formula $C_6H_4(OH)_2$, and with this, as with all di-derivatives of benzene, we have three types, based on the relative positions of the two substituted radicles. Taking $C_6H_4(OH)_2$ as example, we can best explain the three modifications by the graphic formulas given below:

Ortho-dioxybenzene.



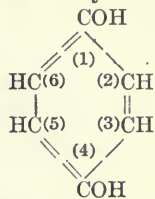
Pyrocatechin or catechol.

Meta-dioxybenzene.



Resorcin or resorcinol.

Para-dioxybenzene.



Hydroquinone or quinol.

It will be seen that the *ortho*-body is one in which the two hydroxyl groups are on adjoining carbon atoms; the *meta*- is the one in which one carbon atom intervenes between the two hydroxyl groups on one side, and three on the other, whereas the *para*-compounds have two carbon atoms intervening between the hydroxyl groups.

Possibly a clearer explanation of this is by numbering the carbon atoms as above, and saying that in the *ortho*-body the hydroxyl groups are attached to carbon atoms number one and two; in the *meta*-, the hydroxyl groups are attached to carbon atoms number one and three; and in the *para*-bodies, the hydroxyl groups are attached to carbon atoms number one and four.

It is hardly necessary to note that this numbering is purely arbitrary, and that a benzene in which two radicles are attached to carbon atoms four and five would be as much *ortho*- as is that attached to one and two as given above.

Of the three diatomic phenols having the formula $C_6H_4(OH)_2$, the *ortho*-compound is called pyrocatechin; the *meta*-, resorcin, and the *para*-, hydroquinone.

Pyrocatechin, obtained by destructive distillation of catechin, is a constituent of catechu, U. S. P. 1890. It is of no pharmaceutic value. The methyl ether of same, however, is guaiacol.

GUAIACOL. Guaiacol.



One of the chief constituents [$C_6H_4(OH)(OCH_3)$ 1:2] of creosote, the product from beechwood tar, obtained by collecting and purifying the fraction of creosote boiling between 200° and 205° C. (392° and 401° F.); or prepared synthetically from either catechol by methylating, or from *ortho*-anisidin by diazotizing and boiling. Guaiacol should be preserved in amber-colored bottles, protected from light.

A colorless, crystalline solid, melting at 28.5° C. (83.3° F.), or a colorless, refractive liquid, boiling at 205° C. (401° F.), having an agreeable aromatic odor.

Specific gravity of liquid: 1.140 at 25° C. (77° F.).

Soluble in 53 parts of water at 25° C. (77° F.) and in alcohol and ether in all proportions; soluble in acetic acid and in 1 part of glycerin.

The addition of ferric chloride T.S. to an alcoholic solution of Guaiacol (1 in 100) causes an immediate blue color, changing to emerald-green, and finally becoming yellowish.

If 2 Cc. of Guaiacol be shaken with 4 Cc. of petroleum benzin, the mixture should separate, on standing, into two distinct layers. Turbidity or failure to separate into layers indicates the presence of impurities.

One Cc. of Guaiacol should dissolve in 2 Cc. of sodium hydroxide T.S., when heated, and, on cooling, the mixture should congeal to a white mass. Coloration or failure to congeal indicates the presence of impurities. The white mass thus obtained should form a clear solution with 20 volumes of water (turbidity indicates *oily hydrocarbons*).

One Cc. of Guaiacol, when added to 10 Cc. of concentrated sulphuric acid, should develop a pure yellowish color in the liquid (a reddish color indicating *creosote*).

Tests for Identity.—Given above.

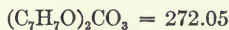
Impurities.—Oily hydrocarbons and creosote. Details above.

This substance can be made synthetically by cooking pyrocatechin with a mixture of potassa and potassium methyl sulphate, although it is usually prepared commercially by the rectification of creosote.

Guaiacol is one of our most valuable internal antiseptics, and for this purpose is used both in tuberculosis and also in intestinal troubles.

Dose.—0.5 Cc. (8 minims).

GUAIIACOLIS CARBONAS. Guaiacol Carbonate.



A guaiacol derivative $[(C_6H_4(OCH_3)O)_2CO]$, obtained by the action of carbonyl chloride upon sodium guaiacolate.

White, crystalline powder, of neutral reaction, almost tasteless and odorless.

Insoluble in water; soluble in 48 parts of alcohol, 1.5 parts of chloroform, and 13 parts of ether at 25° C. (77° F.); readily soluble in hot alcohol and benzene; slightly soluble in glycerin and fatty oils.

When heated between 84° and 87° C. (183.2° and 188.6° F.) it fuses.

Guaiacol Carbonate is at once decomposed when treated with alcoholic potassium hydroxide T.S., and from the solution so obtained guaiacol may be separated on the addition of an acid.

An alcoholic solution of Guaiacol Carbonate should not yield a bluish-green color on the addition of ferric chloride T.S. (absence of *free guaiacol*).

Tests for Identity.—Given above.

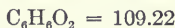
Impurities.—Free guaiacol. Details above.

Remarks.—This solid derivative of guaiacol is known as *duotal*. Its use is similar to guaiacol, over which it possesses the advantage of being less irritating.

Dose.—1 Gm. (15 grains).

Benzozol, $C_6H_4 \begin{smallmatrix} \text{OCH}_3 \\ \text{OCOC}_6H_5 \end{smallmatrix}$, is the benzoic acid ester of guaiacol, and is used like it.

RESORCINOL. Resorcinol.



[RESORCINUM, PHARM. 1890]

A diatomic phenol [metadihydroxybenzene, $C_6H_4(OH)_2$ 1:3], obtained usually by the reaction of fused sodium hydroxide upon sodium metabenzenedisulphonate. Resorcinol should be kept in dark amber-colored vials.

Colorless, needle-shaped crystals, having a faint, peculiar odor and a sweetish, followed by a bitter, taste. It acquires a pinkish tint on exposure to light and air.

Soluble in 0.5 part of water at 25° C. (77° F.), slightly more soluble in alcohol; very soluble in boiling water or in boiling alcohol; also readily soluble in ether and glycerin; very slightly soluble in chloroform, carbon disulphide, and benzene.

When heated to 119° C. (246.2° F.) it melts, and at a higher temperature is completely volatilized. It boils at 276.5° C. (529.7° F.) and is slightly volatile in a current of steam.

Its aqueous solution is neutral or only slightly acid to litmus-paper.

On adding a few drops of ferric chloride T.S. to 10 Cc. of an aqueous solution of Resorcinol (1 in 200), the liquid assumes a bluish-violet color, changing to brownish-yellow on the addition of ammonia water (distinction from *catechol* and *quinol*).

Lead acetate T.S. should produce no precipitate when added to an aqueous solution of Resorcinol (distinction from, and absence of, *catechol*).

If 0.1 Gm. of Resorcinol be dissolved in 1 Cc. of potassium hydroxide T.S. and a drop of chloroform added, the mixture upon being heated will assume an intense crimson color. If a slight excess of hydrochloric acid be then added, the color will change to a pale straw-yellow.

On cautiously heating 0.05 Gm. of Resorcinol with 0.1 Gm. of tartaric acid and 10 drops of concentrated sulphuric acid, a thick carmine-red liquid will be formed, becoming pale yellow when diluted with water.

A concentrated aqueous solution of Resorcinol (1 in 2) should be colorless (absence of *empyreumatic bodies*), and when gently heated should not emit the odor of *phenol*.

Tests for Identity.—Given above.

Impurities.—*Catechol*, *quinole*, *empyreumatic bodies*, *phenol*. Details given above.

This body, metadihydroxybenzene, was discovered by Hlasiwicz in 1864, who obtained it by fusing galbanum with potassa. It can be made in the same way from other gum-resins, such, for example, as *asafetida*, and is now prepared synthetically by fusing metadisulphonbenzene, $C_6H_4(SO_3H)_2$, with potassa.

Resorcinol occurs in white, feathery crystals, turning pinkish on exposure to air; soluble in water, alcohol, and ether. It is used chiefly externally in some forms of skin troubles because of its antiseptic action. Of late it has had considerable vogue as a cure for dandruff. When taken internally, it possesses antipyretic properties.

Dose.—125 milligrammes (2 grains).

Several derivatives have been used in late years in medicine.

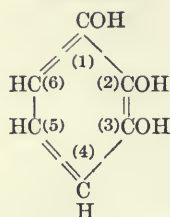
Thioresorcin, $C_6H_4O_2S_2$, was suggested as an iodoform substitute, but has been abandoned on account of its harmful action.

Resopyrin represented a fused mass of resorcinol with antipyrin, and was used as an antipyretic.

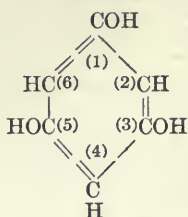
Resorcinol is made by fusing iodoform with resorcin. The name of this is unfortunate, inasmuch as the word resorcinol is the correct modern appellation for resorcin itself.

Hydroquinine is the paradioxy-derivative of benzene, and is obtained by oxidation of cinchonic acid. It is also obtained by hydrolysis of the glucoside arbutin (p. 709). It has practically no medicinal application, although very largely used in photography.

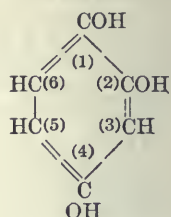
Triatomic phenols of benzene have the formula $C_6H_3(OH)_3$. As there were three diatomic phenols, so there are three bodies with the formula $C_6H_3(OH)_3$, and the graphic formulas of these are given below:

Vicinal trioxy-
benzene.

Pyrogallol.

Symmetric trioxy-
benzene.

Phloroglucin.

Asymmetric trioxy-
benzene.

Oxyhydroquinone.

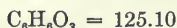
As will be noticed, in the vicinal form, the three hydroxyl groups are on adjoining carbon atoms; in the symmetric, the hydroxyl groups are each separated from the other by an interposed carbon atom; while in the asymmetric two are on adjoining carbon atoms and the other is separated by an interposed carbon atom.

In explaining it in the numeric way, in the vicinal the hydroxyl groups are on carbon atoms 1, 2, and 3; in the symmetric, they are on carbon atoms 1, 3, and 5, and in the asymmetric they are on carbon atoms 1, 2, and 4.

As the classification for the ortho-, meta-, and para-compounds applies to all di-derivatives of benzene, so also the classification of the vicinal, symmetric, and asymmetric applies to all tri-derivatives of benzene.

As noted above, the three tribasic phenols are called pyrogallol, phloroglucin, and oxyhydroquinone respectively.

PYROGALLOL. Pyrogallol.



A triatomic phenol [$\text{C}_6\text{H}_3(\text{OH})_3$, 1:2:3], obtained chiefly by carefully heating gallic acid. Pyrogallol should be kept in dark amber-colored bottles.

Light, white laminæ or fine needles, odorless and having a bitter taste; acquiring a grayish tint on exposure to air and light.

Soluble in 1.6 parts of water, 1 part of alcohol, and in 1.1 part of ether at 25°C . (77°F .); very soluble in boiling water and in boiling alcohol.

When heated to 132°C . (269.6°F .) Pyrogallol melts and sublimes unchanged. When ignited it is consumed, leaving no residue.

The freshly prepared aqueous solution is neutral to litmus-paper and colorless, but gradually acquires by exposure to the air a brown color and an acid reaction, due to absorption of oxygen; the absorption of oxygen and change of color take place very rapidly if the solution contains an alkali hydroxide.

The aqueous solution of Pyrogallol (1 in 10) reduces solutions of the salts of silver, gold, and mercury even in the cold.

When freshly prepared, 1 Cc. of the aqueous solution (1 in 20) is colored brownish-red by a few drops of ferric chloride T.S., and this color is changed to deep bluish-black on the addition of 1 or 2 drops of ammonia water. A bluish-black color is also produced in the aqueous solution of Pyrogallol by freshly prepared ferrous sulphate T.S.

Remarks.—Pyrogallol is what is commercially called pyrogallic acid, and was discovered by Scheele in 1786. It is made by strongly heating gallic acid, hence the name, pyrogallic acid; the prefix, *pyro*, as explained on p. 459, communicating the idea that the body has been produced by strong heat.

As will be explained under gallic acid, the heating of that product causes the loss of CO_2 , with formation of the official pyrogallol.

Pyrogallol is in light white needles, becoming gray or darker on exposure to air and light. It is soluble in 1.6 parts water and one part alcohol; melts at 141°C ., subliming at the same time.

Pyrogallic acid is one of the most valuable reducing agents, having such strong affinity for oxygen that one volume of $2\frac{1}{2}$ per cent. alkaline solution absorbs 14 volumes of the gas, and for this reason it is used as an absorber in gas analysis. Its reducing action makes it valuable as a developer in photography, and it has of late been considerably used as one of the constituents of that class of hair-dyes producing auburn or chestnut tints. These dyes consist of two solutions, one of either copper or nickel salts, and the other, pyrogallol, and when the hair is moistened with the solution of metallic salt and then treated with pyrogallol solution, a literal "copper plating" takes place.

Pyrogallol is used externally in skin troubles, but should be applied only under the direction of skilled physicians, as it is decidedly irritating in action.

Phloroglucin, $\text{C}_6\text{H}_3(\text{OH})_3$ 1 : 3 : 5, is made by fusing kino with potassa or resorcin with soda. It is of no pharmaceutic value save as a test for wood-paper (p. 702).

Oxyhydroquinone, $\text{C}_6\text{H}_3(\text{OH})_3$ 1 : 2 : 4, is an oxidation product of quinine, but of no value either in medicine or pharmacy.

The amido derivatives of benzene are of almost equal importance to the hydroxyl derivatives, $\text{C}_6\text{H}_5\text{NH}_2$ being the well-known aniline.

Aniline was discovered as a distillation product from indigo by Unverdorben in 1826, who obtained the crystalline chloride and called it "*crystallin*." Its presence in coal-tar was demonstrated by Runge in 1824, who, not noticing its identity with "*crystallin*" of the previous investigator, called it "*kyanol*."

Fritzsche investigated the coal-tar derivative in 1841, found it identical with "*crystallin*," and called it aniline, from the Portuguese name for indigo, "*anil*." The chemistry of the subject was not made perfectly clear until Hoffman's investigations of 1843, and the further researches of this master gave rise to our present dye-stuff industry, with its enormous capital and its far-reaching influence.

While the first aniline dye-stuff, mauvein, was manufactured by Perkin in 1856, it was not until 1864 that the manufacture of aniline reached a commercial basis, and in that year the first aniline factory was established. While aniline itself is a constituent of coal-tar, it has been found simpler to convert the benzene distillate of coal-tar into the body by first changing same into nitrobenzene, $\text{C}_6\text{H}_5\text{NO}_2$, and reducing this to aniline, $\text{C}_6\text{H}_5\text{NH}_2$, by treating with hydrochloric acid and iron filings. The product of the reduction is then distilled with steam and purified by repeated distillation if necessary.

Aniline is a straw-colored liquid possessing a peculiar aromatic odor, somewhat similar to benzene; soluble in 31 parts of water and is neutral to litmus-paper. It possesses valuable solvent properties—dissolving indigo, for instance. Its reactions are manifold, they leading to that exceedingly valuable class of compounds, the dye-stuffs,

which at present have assumed such proportions as to make their study a science in itself, and far beyond the scope of this work.

Among these aniline dye-stuffs one has proved of sufficient medical value to be accorded pharmacopœial recognition.

METHYLTHIONINÆ HYDROCHLORIDUM. Methylthionine Hydrochloride. Methylene Blue.



Tetramethylthionine Hydrochloride, obtained by the action of hydrogen sulphide upon an oxidation product of para-amido-dimethyl-aniline.

A dark green, crystalline powder, or in the form of prismatic crystals having a bronze-like lustre.

It is readily soluble in water and somewhat less readily in alcohol, the solutions having a deep blue color.

The addition of hydrochloric acid to its aqueous solution changes the color to a lighter shade of blue.

The addition of sodium hydroxide T.S. to the aqueous solution changes the color to a purplish shade, and if added in excess, produces a precipitate having a dull violet color.

The dry powder dissolved in sulphuric acid containing powdered zinc produces a solution which, upon standing, is gradually decolorized.

Two Gm. of Methylthionine Hydrochloride, when ignited, should leave not more than 0.008 Gm. of residue, which should be free from zinc oxide (absence of commercial dye, and other mineral impurities).

Tests for Identity.—Given above.

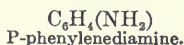
Impurities.—Commercial methylene-blue and mineral impurities. Details above. Arsenic. Details in Part V.

Remarks.—The manufacture of methylene-blue opens up the complex question of synthetic aniline dyes. Aniline (Formula I) can be converted into paraphenylenediamine (Formula II), and this in turn can be methylated to dimethylparaphenylenediamine or para-amidodimethyl aniline (Formula III). When this substance is treated with hydrogen sulphide and ferric chloride, two molecules condense and add sulphur (Formula IV), forming one molecule of methylene-blue (Formula V).

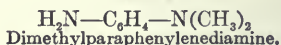
FORMULA I.



FORMULA II.



FORMULA III.

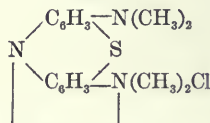


FORMULA IV.



Condensation of dimethylparaphenylenediamine (ammonia splits off).

FORMULA V.



Methylene-blue.

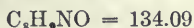
The commercial methylene-blue is a double salt of the pure dye with zinc chloride, hence must not be used. This explains the test for its presence given above.

Methylene-blue is used as an antiseptic in gonorrhea. It is very disagreeable to handle, because it stains everything with which it comes in contact a tenacious blue.

Dose.—250 milligrammes (4 grains).

Aniline is little used in pharmacy, the only derivatives of value in medicine being acetanilide and a few of its congeners.

ACETANILIDUM. Acetanilide.



The monacetyl derivative [$\text{C}_6\text{H}_5\text{NH}(\text{CH}_3\text{CO})$] of aniline.

Colorless, shining, micaceous, crystalline laminae, or a crystalline powder; odorless, having a slightly burning taste, and permanent in the air.

Soluble in 179 parts of water and in 2.5 parts of alcohol at 25° C. (77° F.); in 18 parts of boiling water and in 0.4 part of boiling alcohol; also soluble in 12 parts of ether and 5 parts of chloroform at 25° C. (77° F.).

When heated to 113° C. (235.4° F.) Acetanilide melts, and at 295° C. (563° F.) it boils without decomposition.

Upon ignition it is consumed without leaving a weighable residue.

Solutions of Acetanilide in simple solvents are neutral to test-paper.

If 0.5 Gm. of Acetanilide be agitated with 5 Cc. of colorless sulphuric acid in a clean test-tube, it dissolves without imparting color to the liquid.

On heating 0.1 Gm. of Acetanilide with 5 Cc. of concentrated solution of potassium hydroxide (1 in 4), the characteristic odor of aniline becomes noticeable. On now adding 1 Cc. of chloroform, and again heating, the disagreeable odor of phenyl isocyanide (a poisonous product) is evolved (distinction from *methyl-acetanilide* or *antipyrine*).

On boiling 0.1 Gm. of Acetanilide for several minutes with 2 Cc. of hydrochloric acid, a clear solution results, which, when mixed with 3 Cc. of an aqueous solution of phenol (1 in 20), and afterward with 5 Cc. of a filtered, saturated solution of chlorinated lime, acquires a brownish-red color, becoming deep blue upon supersaturation with ammonia water.

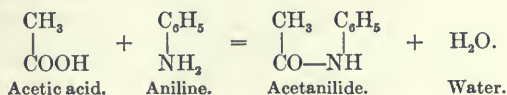
On heating 0.1 Gm. of Acetanilide with 10 Cc. of water, filtering the solution when cold, and adding bromine T.S., drop by drop, to the filtrate, a whitish precipitate of *parabromacetanilide* is formed (distinction from *antipyrine* or *acetphenetidin*).

A cold saturated, aqueous solution of Acetanilide added to ferric chloride T.S. should not affect the color of the latter (absence of *aniline salts* and *various allied substances*).

Tests for Identity.—Given above.

Impurities.—Methylacetanilide, antipyrine, acetphenetidin, aniline salts. Details above.

Remarks.—This body, commonly called *antifebrin*, represents aniline in which one of the hydrogen atoms is replaced by the acetyl ($\text{C}_2\text{H}_3\text{O}$) group. It is made by heating aniline with glacial acetic acid, the reaction being—



Acetanilide, or *phenyl acetamide*, as it is sometimes called, occurs in white, shining lamina or in crystalline powder, sparingly soluble in alcohol, melting at 113° C. It, like aniline, responds to the isonitrile reaction (p. 642).

Acetanilide is one of the most valuable antipyretics, although, like all the modern synthetics of this character, it should be used with caution. The indiscriminate use of these products has caused great harm, being particularly dangerous to persons with weak hearts. It is given in doses of four grains, either in powder or dissolved in brandy, although the latter method of administration is hardly advisable, as the two products are physiologically antagonistic. An excess of

acetanilide produces symptoms of coma and collapse, and the best antidote is copious drafts of brandy.

Dose.—250 milligrammes (4 grains).

ACETANILIDE DERIVATIVES AND SUBSTITUTES

Bromacetanilide is formed when one of the hydrogen atoms of the benzene nucleus is replaced by bromine. It is an antipyretic and antiseptic. Dose, 1 to 8 grains.

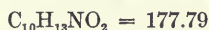
Benzanilide.—In this the benzoyl (C_6H_5CO) group is substituted for the acetyl group in the acetanilide.

Exalgin is methyl acetanilide, or, more properly speaking, acetotoluidide, and is made by heating toluidin ($C_6H_4<\begin{smallmatrix} CH_3 \\ NH_2 \end{smallmatrix}$) with glacial acetic acid.

Antikamnia is a mixture of acetanilide, caffeine, tartaric acid, and sodium bicarbonate. A combination of this character is recognized in the pharmacopœia as Compound Powder of Acetanilide (p. 301).

Antinervine is a compound consisting of 50 per cent. acetanilide, 25 per cent. each ammonium bromide and salicylic acid.

ACETPHENETIDINUM. Acetphenetidin.



A phenol derivative [Acetparaphenetidin, $C_6H_4(OC_2H_5).NH.CH_3.CO$ 1:4], the product of acetylation of para-amidophenetol.

White, glistening, crystalline scales or fine crystalline powder, odorless and tasteless.

It is soluble in 925 parts of water, 12 parts of alcohol, 63 parts of ether, and 20 parts of chloroform at 25° C. (77° F.); in 70 parts of boiling water and in 2 parts of boiling alcohol.

Heated to between 134° and 135° C. (273.2° and 275° F.) it melts, and at a higher temperature burns without leaving a weighable residue.

It dissolves without color in sulphuric acid, but if shaken with nitric acid it is colored yellow, which color persists when heated.

If 0.1 Gm. of Acetphenetidin be boiled for one minute with 1 Cc. of concentrated hydrochloric acid and the solution diluted with 10 Cc. of water, cooled and filtered, it should yield on addition of 3 drops of an aqueous solution of chromium trioxide (1 in 30) a ruby red color.

On heating 0.1 Gm. of Acetphenetidin with 5 Cc. of a concentrated solution of potassium hydroxide (1 in 4), the odor of aniline should not be perceptible.

If 0.1 Gm. of Acetphenetidin be boiled with 10 Cc. of water it should yield a solution which, when cooled and filtered, should not become turbid upon the addition of bromine T.S. in slight excess (absence of *acetanilide*).

If 0.1 Gm. of Acetphenetidin be boiled for one minute with 3 Cc. of solution of sodium hydroxide (1 in 2), the solution cooled, and then agitated with 5 Cc. of a solution of chlorinated soda, there should be produced a clear yellow liquid, and not a purplish-red or brownish-red cloudy liquid or precipitate (absence of *acetanilide*).

A mixture of 0.3 Gm. of Acetphenetidin with 1 Cc. of 90 per cent. alcohol should not acquire a red tint when diluted with three times its volume of water and boiled with one drop of tenth-normal iodine V.S. (absence of *paraphenetidin*).

Tests for Identity.—Given above.

Impurities.—Acetanilide, paraphenetidin.

Remarks.—This body, commonly known as *phenacetine*, has the formula $C_6H_4<\begin{smallmatrix} OC_2H_5 \\ NHC_2H_5O \end{smallmatrix}$. It represents acetanilide in which one of the hydrogen atoms is replaced by an OC_2H_5 group. Speaking more technically, it represents an ethyl acetyl derivative of para-amidophenol, $C_6H_4<\begin{smallmatrix} OH \\ NH_2 \end{smallmatrix}$, and is made by the reduction of para-nitrophenol, which, in turn, is prepared by treating carbolic acid with cold diluted nitric acid. By treating para-amido-phenol with ethyl iodide,

p-phenetidin, $C_6H_4<\begin{smallmatrix} OC_2H_5 \\ NH_2 \end{smallmatrix}$, is produced, and by treating the latter with glacial acetic acid acetyl phenetidin or phenacetine results.

Phenacetine is a white powder that melts at 134° to 135° C. It is one of the most valuable antipyretics.

Phenacetine is sometimes contaminated with phenetidin. This latter is a dangerous irritant, hence such adulterated phenacetine should not be dispensed. Detection of same is easily accomplished by the pharmacopœial test given above.

Some unscrupulous dealers have adulterated phenacetine with acetanilide, but this can be easily detected by means of the melting-point—phenacetine, 134° to 135° ; acetanilide, 113° —or by two pharmacopœial chemical tests given above.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

As with all the successful modern synthetics, the introduction of phenacetine was followed by a large number of bodies of similar composition. Thus we have *iodophenin*, which represents phenacetine hydrochloride combined with iodine and potassium iodide.

Methacetin, $C_6H_4<\begin{smallmatrix} OCH_3 \\ NHC_2H_5O \end{smallmatrix}$, differs from phenacetine only in having a methyl group instead of the ethyl group of phenacetine, and, like it, is used as an antipyretic.

Hydracetin or *pyrodin*, $C_6H_5-NH-\begin{smallmatrix} | \\ NHC_2H_5O \end{smallmatrix}$, which is acetphenylhydrazin and made similar to acetanilide, save for the use of phenylhydrazin instead of aniline.

Phenocoll hydrochloride, $C_6H_4<\begin{smallmatrix} OC_2H_5 \\ NHC_2H_5NH_2 \end{smallmatrix}$, is made by treating phenetidin with amido-acetic acid, $CH_2<\begin{smallmatrix} NH \\ COOH \end{smallmatrix}$ (glycocoll). Phenacoll hydrochloride occurs in white crystals, soluble in 20 parts of water, and in this way possesses decided advantage over phenacetine, which is soluble in 925 parts of water, as mentioned above. It, like phenacetine, is used as an antipyretic. Strictly speaking, it might more properly be considered under the hydrocarbons other than benzene, as both phenacetine and phenocoll contain more than six carbon atoms. However, as they so closely resemble acetanilide, it was deemed advisable to deviate from the regular order, that these closely similar bodies might be considered together.

CHAPTER XLIV

TOLUENE COMPOUNDS

TOLUENE has the formula $C_6H_5CH_3$. It represents benzene in which one hydrogen is replaced by a methyl group. In this body, therefore, we have a combination of the ring and chain compounds, the methyl group possessing nearly all the reactions of the chain series of hydrocarbons, while the phenyl acts exactly as it did in benzene.

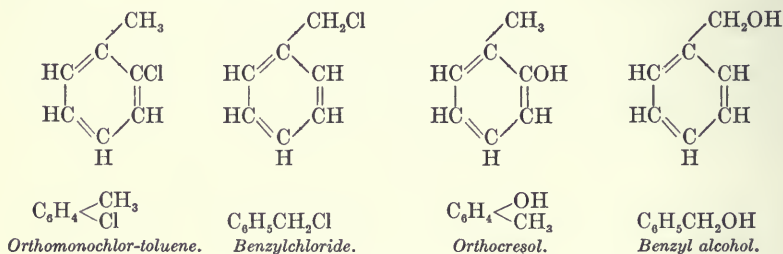
Toluene was first made by dry distillation of rosin by Pelletier and Walter in 1838. Deville (1841) obtained it by distillation of balsam of tolu, and from this circumstance Berzelius named it toluol.

It can be easily made by several reactions; chief among these are, (1) the *Friedel Craft reaction*, in which benzene is treated with methyl chloride in the presence of aluminum chloride, and (2) by the *Fittig*

reaction, in which phenyl bromide is treated with methyl iodide in the presence of metallic sodium, this latter way being a modification of the Wurtz reaction mentioned on p. 634.

These two reactions are mentioned by name because affording means of the manufacture of any of the hydrocarbons of this series. Thus if ethyl chloride is substituted for the methyl chloride in the first reaction, or if ethyl iodide replaces methyl iodide in the second reaction, we would get ethyl benzene, $C_6H_5C_2H_5$.

Most commercial toluene, however, is obtained by the fractional distillation of coal-tar, and is a colorless or amber-colored liquid, of aromatic odor, suggestive of benzene. It has a specific gravity 0.870, and boils at $110^\circ C.$, and is insoluble in water, but freely soluble in alcohol. In its chemical behavior it resembles benzene, on one hand, and methane, on the other, and in the same way forms two distinct lines of hydrocarbon derivatives. Thus, the replacing of one hydrogen of the benzene group by the addition of chlorine gives rise to the compound known as monochlor-toluene, and this body is produced when chlorine is passed into cold toluene. On the other hand, if boiling toluene is treated with chlorine, the halogen replaces one of the hydrogens of the methyl group, forming the body called benzyl chloride. The structural difference between these two bodies is best shown by the graphic formulas given below. These two compounds are mentioned not because of their importance in themselves, but by reason of the possible forms when the respective chlorines are replaced by hydroxyls, in the first case a phenol (cresol) being formed, whereas the benzyl chloride yields benzyl alcohol, which possesses all the characteristics of an ordinary fatty alcohol.



Under appropriate treatment of boiling toluene with chlorine, there is also yielded benzotrichloride, $C_6H_5CCl_3$, and benzal chloride, $C_6H_5CHCl_2$.

The toluene derivatives of pharmaceuic interest are:

Cresol.....	$C_6H_4 \begin{smallmatrix} \text{OH} \\ \text{CH}_3 \end{smallmatrix}$
Creosol.....	$C_6H_3 \begin{smallmatrix} \text{CH}_3 \\ \text{OCH}_3 \\ \text{OH} \end{smallmatrix}$
(Creosote, U. S. P.)	
Benzyl alcohol.....	$C_6H_5CH_2OH$
Benzaldehyde.....	C_6H_5CHO
(Oil bitter almond)	
Benzoic acid.....	C_6H_5COOH

Saccharin.....	$C_6H_4 \begin{matrix} SO_2 \\ CO \end{matrix} > NH.$
Salicylic acid.....	$C_6H_4 \begin{matrix} COOH \\ OH \end{matrix}$
Salol.....	$C_6H_4 \begin{matrix} OH \\ COOC_6H_5 \end{matrix}$
Vanillin.....	$C_6H_3 \begin{matrix} OH \\ OCH_3 \\ CHO \end{matrix}$
Gallic acid.....	$C_6H_2 \begin{matrix} (OH)_3 \\ COOH \end{matrix}$
Tannic acid.	

The *cresols*, $C_6H_4 \begin{matrix} OH \\ CH_3 \end{matrix}$, are the monobasic phenols of toluene, and, like all di-derivatives of benzene, are known in the *ortho*-, *meta*-, and *para*- forms.

CRESOL. Cresol.

$$C_7H_7.OH = 107.25$$

A mixture [$C_6H_4(CH_3)OH$] of the three isomeric Cresols obtained from coal-tar, freed from phenol, hydrocarbons, and water. It should be preserved in amber-colored bottles, protected from light.

A colorless or straw-colored refractive liquid, having a phenol-like odor, and turning yellowish-brown on prolonged exposure to light.

Specific gravity: 1.032 at 25° C. (77° F.).

Cresol is soluble in 60 parts of water at 25° C. (77° F.); miscible in all proportions with petroleum benzin, benzene, alcohol, ether, and glycerin; miscible with alkali hydroxide solutions.

It boils from 195° to 205° C. (383° to 401° F.).

If 1 Cc. of Cresol be mixed with 1 Cc. of an aqueous solution of sodium hydroxide (1 in 10), it should dissolve with no appreciable liquid residue (absence of or limit of hydrocarbons).

If 1 Cc. of Cresol be mixed with 1 Cc. of glycerin, a clear solution should be produced, from which, on the addition of 1 Cc. of water, the Cresol should completely separate (absence of, and distinction from, *phenol*).

Tests for Identity.—Given above.

Impurities.—Hydrocarbons, phenol. Details above.

Remarks.—As mentioned in the official definition, commercial cresol (or *cresylic acid*, as it is frequently called) is a mixture of the three cresols mentioned above. It has been accorded pharmacopœial recognition because a constituent of the new official, *compound solution of cresol*. It is sometimes used internally as an intestinal antiseptic.

Dose.—0.05 Cc. (1 minim).

LIQUOR CRESOLIS COMPOSITUS. Compound Solution of Cresol.

<i>Recipe</i> .—Cresol, five hundred grammes.....	500 Gm.
Linseed Oil, three hundred and fifty grammes.....	350 Gm.
Potassium Hydroxide, eighty grammes.....	80 Gm.
Water, a sufficient quantity,	

To make one thousand grammes.. 1000 Gm.

Dissolve the Potassium Hydroxide in *fifty grammes* of Water in a tared dish, add the Linseed Oil, and mix thoroughly. Then add the Cresol and stir, until a clear solution is produced, and finally sufficient Water to make the finished product weigh *one thousand grammes*.

Remarks.—This is a 50 per cent. solution of cresol in an alkaline linseed oil soap solution, and is a product therapeutically similar to the popular alkaline solutions of coal-tar distillates, such as *creolin*, *lysol*, etc. Like these, it is used as an antiseptic wash or douche.

Losophane, $\text{CH} \begin{smallmatrix} \diagup \text{I}_3 \\ \diagdown \text{CH}_3 \end{smallmatrix} \text{OH}$, is cresol tri-iodide, contains 78.38 per cent. iodine, and is used as an iodoform substitute.

Europhene, $\text{C}_6\text{H}_5 \begin{smallmatrix} \diagup \text{OH} \\ \diagdown \text{C}_4\text{H}_9 \end{smallmatrix}$, is di-isobutylortho-cresol iodide. This name is inserted

merely to show how simple it is to explain in words a graphic formula, a comparison of the formula just given with the name proving the fact. This body contains 28.17 per cent. iodine, and is used like aristol.

Homopyrocatechin, so named because of its similarity to pyrocatechin, is the diatomic phenol of toluene. Its formula is $\text{C}_6\text{H}_3 \begin{smallmatrix} \diagup \text{CH}_3 \\ \diagdown (\text{OH})_2 \end{smallmatrix}$.

Its methyl ester, $\text{C}_6\text{H}_3 \begin{smallmatrix} \diagup \text{CH}_3 \\ \diagdown \text{OCH}_3 \end{smallmatrix} \text{OH}$, is known as *creosol*, and is of interest as a constituent of creosote.

CREOSOTUM. Creosote.

A mixture of phenols and phenol derivatives, chiefly guaiacol and creosol, obtained during the distillation of wood-tar, preferably of that derived from the beech, *Fagus silvatica* Linné or *Fagus ferruginea* Aiton (Fam. *Fagaceæ*).

An almost colorless, yellowish (not pinkish), highly refractive, oily liquid, having a penetrating, smoky odor, and a burning, caustic taste; it should not become brown in color on exposure to light.

Specific gravity not below 1.072 at 25° C. (77° F.).

Its solution in about 140 parts of water at 25° C. (77° F.) is not perfectly clear. With 120 parts of hot water it forms a clear liquid, which, on cooling, becomes turbid from the separation of minute oily drops (distinction from, and absence of, both *phenol* and so-called "*coal-tar creosote*"). The filtrate from these separated oily globules yields a reddish-brown precipitate with bromine T.S. (distinction from *phenol* and so-called "*coal-tar creosote*," both of which yield white precipitates). Soluble in all proportions in absolute alcohol, ether, chloroform, carbon disulphide, acetic acid, and fixed and volatile oils.

When distilled, most of it comes over between 200° and 220° C. (392° and 428° F.). When cooled to -20° C. (-4° F.), it becomes gelatinous, but does not solidify (difference from *phenol*). It is inflammable, burning with a luminous, smoky flame.

Creosote is neutral or only faintly acid to litmus-paper.

On stirring together equal volumes of Creosote and collodion in a dry test-tube, no permanent coagulum should form (difference from *phenol* and so-called "*coal-tar creosote*," and limit of the former).

If 1 volume of Creosote be mixed with 1 volume of 95 per cent. glycerin, a clear mixture will result, from which a creosotic layer, equal to or greater in volume than the Creosote employed, will separate on the addition of one-fourth volume of water (difference from, and limit of, *phenols*).

On adding to 10 Cc. of a saturated aqueous solution of Creosote 1 drop of ferric chloride T.S., the liquid develops a clear violet-blue color, which is very transient; it then clouds almost instantly, the color passing rapidly from a grayish-green into a muddy-brown, with finally the formation of a brown precipitate (difference from *phenol* and so-called "*coal-tar creosote*," and limit of the former).

If 1 Cc. of Creosote be mixed with 10 Cc. of a solution of potassium hydroxide in absolute alcohol (1 in 5), a solid crystalline mass will form (difference from *phenol* and so-called "*coal-tar creosote*," and limit of the former).

On mixing 2 Cc. of Creosote with 10 Cc. of normal sodium hydroxide V.S., a clear, pale yellow liquid results, which remains unclouded on diluting with 50 Cc. of water (absence of *neutral oils*).

If 1 Cc. of Creosote be cautiously and gently shaken with 2 Cc. of petroleum benzin and 2 Cc. of freshly prepared barium hydroxide T.S. until a uniform mixture is produced, upon complete separation three distinct layers are visible, the middle one of which contains the Creosote, unaltered in appearance; while the petroleum benzin should not be blue or muddy, and the aqueous layer should not have acquired a red tint (absence of *cærulignol* and some other *high-boiling constituents of wood-tar*).

Tests for Identity.—Given above.

Impurities.—Phenol, coal-tar creosote, neutral oils, *cærulignol*. Details above.

This body was discovered by Reichenbach in 1830. The pharmacopœia defines it “as a mixture of phenols, . . . chiefly guaiacol and creosol.”

Of these two bodies, guaiacol was described on p. 763, while the origin of creosol has just been given.

Creosote is obtained either from pyroligneous acid (p. 645) (the empyreumatic liquid obtained by the destructive distillation of oak wood) or from beechwood tar. As the last contains 25 per cent. creosote, while pyroligneous acid yields only 1 per cent., the former source of creosote is preferred.

The separation of creosote from the tar is a laborious process. The tar is distilled, the half coming over first is allowed to stand, when it separates into three layers. The lowest layer is the one containing the creosote.

The creosote layer is separated from the others, and saturated with sodium carbonate to neutralize acetic acid present, and is then distilled. This distillate yields two layers, the lower of which contains the creosote, and this is purified by treatment with phosphoric acid. After neutralizing the acid with ammonia and washing the oil with water, it is again distilled, the distillate then consisting of creosote and eupion; the latter is a body named by Reichenbach, and consists chiefly of pentane. The creosote is freed from eupion by treatment with solution of potassa, which saponifies it, while eupion floats on top and can be separated. The saponified creosote is then treated with sulphuric acid, and the free creosote rectified by repeated distillations.

Creosote is a straw- to amber-colored liquid, possessing a characteristic tarry color, and somewhat burning taste. It has a specific gravity of 1.072, is soluble in 150 parts of water, although liquid is cloudy, and is soluble in alcohol, ether, and chloroform. As already noted, a large amount of the creosote in commerce is nothing but an impure form of carbolic acid, and special emphasis should be laid on the fact that such “coal-tar creosote” should never be used for internal administration; nor is there any excuse for ignorantly dispensing same, inasmuch as the pharmacopœia offers three distinct tests whereby the difference between creosote and carbolic acid may be recognized, viz., the collodion test, glycerin solubility, and the ferric chloride test, all of which were fully described above, as well as under Phenol (p. 760).

Creosote is a valuable antiseptic, its preservative action being due to the ease with which it coagulates albumin. This principle is

employed in the so-called "smoking" of meats. The fresh meat is suspended in the vapors of burning wood, when the creosote of the vapors attack the albumin on the outer surface of the ham, forming an impermeable and antiseptic layer. To secure best results in smoking, beechwood should be used, though this is seldom the case.

Several preparations have been placed on the market under the name of "liquid smoke," and the like, and intended to be applied with a brush, consisting of various modifications of creosote or of crude pyroligneous acid, and this method of preserving meat is by no means as satisfactory as by the old process of smoking.

Benzyl alcohol, $C_6H_5CH_2OH$, is the aromatic alcohol of toluene, and appears in the balsam of Peru and balsam of tolu, and can be made by treating benzyl chloride with moist silver oxide. It, like all primary alcohols, oxidizes first to aldehyde and then to acid, and in this way differs markedly from the cresols mentioned above, these being phenols, resembling more closely either the tertiary or the secondary alcohols.

Benzyl alcohol has but little pharmaceutic interest, and is mentioned merely because of its oxidation product, benzaldehyde.

BENZALDEHYDUM. Benzaldehyde.



An aldehyde, produced artificially, or obtained from natural oil of bitter almond or other oils, and containing not less than 85 per cent. of pure Benzaldehyde [$C_6H_5 \cdot COH$]. It should be kept in small amber-colored, well-stoppered bottles.

A colorless, strongly refractive liquid, having a bitter-almond-like odor, and a burning, aromatic taste.

Specific gravity: about 1.045 at 25° C. (77° F.).

Sparingly soluble in water (1 in 300); soluble, in all proportions, in alcohol, ether, and fixed and volatile oils.

Boiling-point: 179° to 180° C. (354.2° to 356° F.); optically inactive.

If 10 drops of Benzaldehyde, dissolved in a little alcohol, be shaken with a few drops of a strong solution of sodium hydroxide, then with a little ferrous sulphate T.S., and finally mixed with a slight excess of hydrochloric acid, a blue precipitate should not be produced (absence of *hydrocyanic acid*).

If the looped end of a piece of clean copper wire be held in a non-luminous flame until it glows, then cooled, and the loop dipped into Benzaldehyde, ignited, and held so that the liquid burns outside of the flame, then if the loop be slowly brought in contact with the lower outer edge of the flame, no green tinge should be discernible (absence of *chlorinated products*).

If a small strip of filter-paper folded in the form of a taper and saturated with Benzaldehyde be placed in a small porcelain dish, and a clean beaker, moistened on the inner surface with distilled water, be inverted over the smaller dish immediately after igniting the taper, a part of the products of combustion will be absorbed by the water; if the beaker be then rinsed with a little distilled water and the liquid filtered, the filtrate should yield no turbidity upon the addition of a few drops of silver nitrate T.S. (absence of *chlorinated products*).

Tests for Identity.—Given above.

Impurities.—Hydrocyanic acid, chlorinated products, given above.

Assay.—Details in Part V.

Remarks.—Benzaldehyde, C_6H_5CHO , can be made by the oxidation of the benzylalcohol just mentioned, or by treatment of benzyl chloride with milk of lime. It occurs in nature as the chief constituent of oil of bitter almond. It is used in perfumery, and rarely employed medicinally.

Dose.—0.03 Cc. ($\frac{1}{2}$ minim).

OLEUM AMYGDALÆ AMARÆ. Oil of Bitter Almond.

A volatile oil obtained from Bitter Almond and other seeds containing amygdalin, yielding, when assayed as directed below, not less than 85 per cent. of benzaldehyde, and not less than 2 per cent. nor more than 4 per cent. of hydrocyanic acid. It should be kept in small, well-stoppered, completely filled, amber-colored bottles, protected from light and air.

A clear, colorless or yellow, thin, and strongly refractive liquid, having a peculiar aromatic odor, and a bitter and burning taste.

Specific gravity: 1.045 to 1.060 at 25° C. (77° F.).

Boiling-point: about 180° C. (356° F.). Optically inactive.

Soluble in 300 parts of water at 25° C. (77° F.), in alcohol or ether in all proportions, and in an equal volume of 70 per cent. alcohol; soluble in nitric acid at ordinary temperatures without the evolution of nitrous vapors.

In the fresh state the Oil is neutral to litmus-paper, but when kept for some time it acquires an acid reaction, due to the formation of benzoic acid, which, when isolated and purified, should correspond to the tests given under *Acidum Benzoicum*. Oil of Bitter Almond containing crystals of benzoic acid should not be dispensed.

If 10 drops of the Oil, dissolved in a little alcohol, be shaken with a few drops of sodium hydroxide T.S., also with 2 drops of ferrous sulphate T.S., and 2 drops of ferric chloride T.S., then warmed, and finally mixed with a slight excess of hydrochloric acid, a blue precipitate will be produced (presence of *hydrocyanic acid*).

Tests for Identity.—Given above.

Impurities.—Chlorinated products. Tested like benzaldehyde, except the additional remark that if a slight turbidity results, it should disappear on boiling.

Assay for Benzaldehyde.—Details in Part V.

Assay for Hydrocyanic acid.—Details in Part V.

This is the volatile oil obtained from bitter almond by maceration with water and subsequent distillation.

Bitter almonds yield both fixed and volatile oil, and "sweet almond oil"—the expressed oil of almond—as obtained by expression from both kinds of almonds—bitter as well as sweet (p. 726).

Bitter almond in its dry form contains no volatile oil—no oil of bitter almond. To produce this, it is necessary to mash up the seed and add water, when a chemical reaction occurs between the amygdalin and emulsin present in the seed, one of the products of the reaction being oil of bitter almond.

Amygdalin belongs to the class of substances called glucosides (p. 709), which are bodies that on treatment with a diluted acid or ferment split into glucose and some other body. Emulsin is one of the unformed ferments explained on p. 937, hence a body that can break up amygdalin, as outlined above.

When the amygdalin is split up by the emulsin, glucose, hydrocyanic acid, and oil of bitter almond are the three decomposition products, and only by the breaking up of the amygdalin is the oil developed. In the dry seed the amygdalin and emulsin rest quietly side by side, just as do the sodium bicarbonate and tartaric acid in dry effervescent salts, such as granular magnesium citrate. As soon as the water is added, just as the effervescent salts show energetic chemical action, even so, though marked only by the development of the bitter almond odor, does the emulsin react with the amygdalin.

In obtaining the bitter almond oil the fixed oil is separated by expression. The press cake is then mixed with water at 50° C. for about a day and then distilled. This results in the hydrolysis of the

amygdalin under the influence of the ferment emulsin, as just explained. When using the ferment, great care must be exercised to prevent too great heat, as that causes decomposition of the ferment and its subsequent inactivity. This statement is worth bearing in mind, as there has arisen the erroneous impression that the reason why cold water is used in the wild-cherry preparations is to prevent the evaporation of the hydrocyanic acid. This is only partly true, the real object of avoiding hot water being to prevent decomposition of the ferment employed. From the volatile oil of bitter almond the hydrocyanic acid can be removed by treatment with ferrous sulphate and milk of lime and redistillation of the product with steam. This, however, is not done in the manufacture of the pharmaceutical oil; the pharmacopœia specifying that same shall contain 2 to 4 per cent. hydrocyanic acid, and this fact gives us a means of distinction between the synthetic benzaldehyde, which is free from hydrocyanic acid, and the natural oil of bitter almond. A very large quantity of such oil of bitter almond on the market represents synthetic benzaldehyde, and can be detected as mentioned above. Note in the pharmacopœial description given above that the crystals forming in bottles of the oil consist of benzoic acid.

Do not confound this synthetic oil of bitter almond—benzaldehyde—with the so-called artificial oil of bitter almond or *oil of mirbane*. This has nothing to do with the almond, being $C_6H_5NO_2$, nitrobenzene. Its unfortunate synonym is due to the fact that its odor suggests that of bitter almond.

Dose.—0.03 Cc. ($\frac{1}{2}$ minim).

AMYGDALA AMARA. Bitter Almond.

The ripe seed of *Prunus Amygdalus* Stokes, var. *amara* De Candolle (Fam. *Rosaceæ*).

Constituents.—Fixed oil, 45 per cent.; amygdalin, 2 to 3 per cent.; emulsin; asparagin; proteid.

Remarks.—The formation of the volatile oil, which does not exist in the dry seeds, but which is formed by the action of the emulsion on amygdalin, has been fully described just above.

Medical Properties.—Sedative. Used largely as a flavor. Seed rarely employed in medicine, the volatile oil being preferable.

PRUNUS VIRGINIANA. Wild Cherry.

The bark of *Prunus serotina* Ehrhart (*Prunus virginiana* Miller) (Fam. *Rosaceæ*), which should be collected in autumn and carefully dried and preserved.

Constituents.—Starch, resin, tannin, volatile oil; amygdalin and emulsin; fluorescent principle.

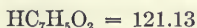
Remarks.—The dry bark contains neither oil nor hydrocyanic acid, but, like bitter almond, these principles are produced when the bark is moistened, and, therefore, constitute the medicinal activity of the drug. A simple test for wild cherry is that it is the only official bark which, when chewed, gives the odor of hydrocyanic acid (or of bitter almond). The ferment emulsin is rendered inactive when treated with hot water, and no oil nor hydrocyanic acid will be developed in

wild cherry when so heated; hence all official preparations of the bark, including the infusion, are made with cold solvents.

Medical Properties.—Tonic, mild sedative (for coughs). Used in form of infusion (p. 215), syrup (p. 197), and fluidextract (p. 253), all of which are official.

Dose.—2 Gm. (30 grains).

ACIDUM BENZOICUM. Benzoic Acid.



An organic acid [$\text{C}_6\text{H}_5\text{COOH}$], obtained from benzoin by sublimation, or prepared artificially. It should be kept in dark amber-colored, well-stoppered bottles, in a cool place.

White or yellowish-white, lustrous scales or friable needles, nearly odorless, or having a slight characteristic odor resembling that of benzoïn, and an acid, pungent taste; somewhat volatile at a moderately warm temperature, and acquiring a yellow color on exposure to light.

Soluble in 281 parts of water and in 1.8 parts of alcohol at 25°C . (77°F .); in 15 parts of boiling water and in 1 part of boiling alcohol; also soluble in 3 parts of ether, 7 parts of chloroform, and readily soluble in carbon disulphide, benzene, fixed and volatile oils; sparingly soluble in petroleum benzin.

Benzoic acid volatilizes freely with the vapor of water. On heating it to 100°C . (212°F .) it begins to sublime. At 121.4°C . (250.5°F .) it melts, and at a higher temperature it is consumed without leaving a residue.

The acid sublimed from benzoin has a lower melting-point, and a greater solubility in water, than that prepared artificially.

An aqueous solution of Benzoic Acid colors blue litmus-paper red.

On heating Benzoic Acid gradually with 3 parts of freshly slaked lime in a dry test-tube, benzene is evolved.

Benzoic Acid is freely soluble in solutions of alkali hydroxides. On carefully neutralizing such a solution, and adding ferric chloride T.S., previously diluted with 2 volumes of water, and neutralized, if necessary, by ammonia water, a flesh-colored precipitate of ferric benzoate is produced.

A solution of Benzoic Acid in pure, cold sulphuric acid, when gently warmed, should not turn darker than light brown; if is then poured into water, the Benzoic Acid should separate as a white precipitate, and the liquid should be colorless (absence of readily carbonizable, organic matters).

If 0.5 Gm. of the Acid and 0.8 Gm. of calcium carbonate be mixed with a little distilled water in a crucible, the mixture dried, gently ignited, and then dissolved in 20 Cc. of distilled water, with the aid of nitric acid in slight excess, and filtered, the addition of silver nitrate T.S. should not produce much more opalescence than is produced by the same quantity of the same reagent in a solution measuring 20 Cc., prepared by dissolving 0.8 Gm. of the same calcium carbonate in distilled water with the aid of nitric acid (absence of more than traces of chlorine).

On warming 0.5 Gm. of the Acid with 5 Cc. of water and 0.5 Gm. of potassium permanganate in a test-tube, loosely stoppered, and placing it in a water-bath heated to about 45°C . (113°F .) for about ten minutes, then tightly stoppering, and cooling the test-tube with cold water, upon removing the stopper, no odor of oil of bitter almond should be discernible (absence of cinnamic acid).

Tests for Identity.—Given above.

Impurities.—Carbonizable matter, chlorine, and cinnamic acid. Details above. Hydrochloric acid. Details in Part V.

Remarks.—Benzoic acid represents the final oxidation product of benzyl alcohol, this yielding benzaldehyde, and it in turn yielding benzoic acid.



Benzoic acid was discovered by Vigeuere in 1608 in benzoin, hence the name. Scheele in 1785 discovered it was a constituent of the

urine, and Liebig and Woehler in 1832 proved its chemical composition. As mentioned above, benzoic acid is a normal constituent of benzoin, and is also an important constituent of balsams; balsams being oleoresins containing benzoic and cinnamic acids.

Benzoic acid can be made from benzene by converting same first into phenyl chloride, C_6H_5Cl , changing this to benzonitril, C_6H_5CN , and treating this with potassium hydroxide, when the potassium benzoate is formed.

As mentioned above, it is also made by the oxidation of either benzylalcohol or benzaldehyde. Commercial benzoic acid, however, is usually manufactured by one of the four following methods:

(a) *Sublimation of benzoin*, as described fully on page 1102.

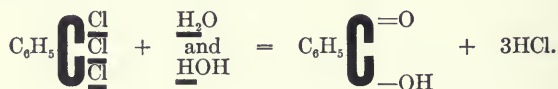
(b) *From Hippuric Acid*.—This body is a constituent of the urine of cattle, and is benzoyl glycocholl, $C_6H_5CONHCH_2COOH$.

On boiling this substance with hydrochloric acid it splits into glycocholl (amido-acetic acid), CH_2COOH , and benzoic acid.

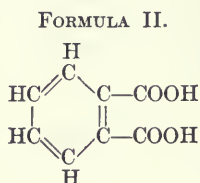
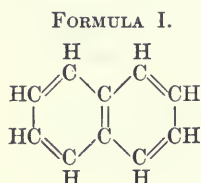
The benzoic acid thus produced is usually mixed with benzoin and resublimed, and when fresh, is an acceptable preparation. In older samples the origin can be readily detected by the odor of stale urine. The benzoic acid of this source was formerly used in considerable quantities under the name of *German benzoic acid*, but at the present time the process has been largely supplanted by a third method of manufacture.

(c) *From Toluene*—A comparison of the formulas of toluene, $C_6H_5CH_3$, and of benzoic acid, C_6H_5COOH , will show that the difference between them is slight; benzoic acid has two atoms of oxygen more and two atoms of hydrogen less, and, therefore, it should be easily prepared by oxidation.

This, however, is not wholly the case, the reaction being rather indirect. Toluol is treated with chlorine, changing it, $C_6H_5CH_3$ into $C_6H_5CCl_3$, and this, when boiled with water, is converted into benzoic and hydrochloric acids, by the following reaction:



(d) *Process of Manufacture from Naphtalin*.—This body is $C_{10}H_8$, or, graphically, as expressed in Formula I below, and on oxidation with nitric acid, it yields phtalic acid, $C_6H_4(COOH)_2$, which is graphically expressed in Formula II.

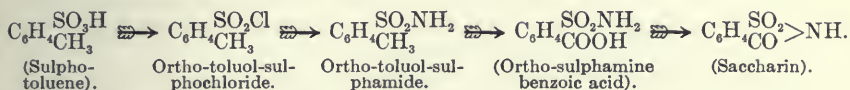


Phthalic acid, on treatment with calcium hydroxide, loses one CO_2 group, and is converted into benzoic acid, $\text{C}_6\text{H}_5\text{COOH}$, or rather into its calcium salt.

Benzoic acid is used as an antiseptic and employed medicinally chiefly in the form of metallic benzoates.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Sulphobenzoic acid, $\text{C}_6\text{H}_4\text{SO}_3\text{H}$, made by treating benzoic acid with sulphuric acid, is mentioned because of its amid-derivative, *saccharin*, $\text{C}_6\text{H}_4\text{SO}_2\text{NH}$, which was discovered by Remsen and Fahlberg in 1879. Its synthesis can best be expressed by successive products of the reaction:



Toluene, when treated with sulphur trioxide, yields sulphotoluene, $\text{C}_6\text{H}_4\text{SO}_3\text{H}$. This yields successively the products given above, the end-product being saccharin, which is official as—

BENZOSULPHINIDUM. Benzosulphinide. Saccharin.



The anhydride of ortho-sulphamide-benzoic acid (benzoyl sulphonic-imide).

A white, crystalline powder, nearly odorless, having an intensely sweet taste even in dilute solutions.

Soluble in 250 parts of water and in 25 parts of alcohol, and but slightly soluble in ether or chloroform at 25°C . (77°F .); soluble in 24 parts of water at 100°C . (212°F .). It is easily soluble in ammonia water, in alkali hydroxide solutions, and in a solution of sodium bicarbonate, with the evolution of carbon dioxide.

Heated between 219° and 220°C . (426.2° and 428°F .) it melts, and at higher temperatures burns with an odor of oil of bitter almond, without leaving a weighable residue (absence of *inorganic impurities*).

If 0.2 Gm. of Benzosulphinide be dissolved with agitation in 10 Cc. of pure sulphuric acid, and the solution kept at a temperature of from 48° to 50°C . (118.4° to 122°F .), on a water-bath, it should not, within ten minutes, show a brown color (absence of *carbohydrates*).

If 0.2 Gm. of Benzosulphinide be dissolved in 5 Cc. of potassium hydroxide T.S., the solution should be clear, and not become colored, even on prolonged heating (absence of *glucose*).

A similar solution mixed with 5 Cc. of alkaline cupric tartrate V.S. should not, on heating, deposit any red cuprous oxide (absence of *glucose* or *milk-sugar*).

If to a hot aqueous solution of Benzosulphinide, ferric chloride T.S. be added, drop by drop, no precipitation or violet color should appear (absence of *benzoic* or *salicylic acids*).

Tests for Identity.—Given above.

Impurities.—Carbohydrates, glucose, milk-sugar, benzoic and salicylic acids. Details above.

Remarks.—Saccharin is in the form of white powder, sparingly soluble in water, and possessing a taste that has been graphically described as being “so sweet that it is bitter” (being 300 times sweeter than sugar), and in order to obtain its full sweetening power it is

...ary that it be diluted. Administered as a sweetener, it is usually combined with alkali, such as sodium bicarbonate, and the saccharine tablets intended for use by diabetic patients in place of sugar are combined with the required quantity of alkali before compression. The addition of alkali is in order to render them more soluble.

As noted in the official description just given, saccharin is only sparingly soluble in water. The sodium compound, on the other hand, is very soluble, although not so powerful an antiseptic.

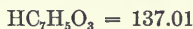
Saccharin is by no means a harmless agent, and its indiscriminate use as a sweetener should be deprecated. For diabetic patients it is a boon, but, on the other hand, for sweetening foods in order to spare the cost of the sugar its use should be strongly condemned.

It is of interest to note that saccharin possesses valuable antiseptic properties, the researches of Miller showing that saccharin ranked in activity next to bichloride of mercury as an agent for destroying the bacteria of the mouth.

Professor Miller devised a saccharin mouth-wash containing saccharin, an astringent substance like krameria, blended with aromatics, and products similar to this are among the most popular of the modern mouth-wash preparations.

Dose.—200 milligrammes (3 grains).

ACIDUM SALICYLICUM. Salicylic Acid.



A monobasic organic acid [$\text{C}_6\text{H}_4(\text{OH})\text{COOH}$ 1:2], existing naturally in combination in various plants, but generally prepared synthetically from phenol.

Light, fine, white, prismatic needles, or a bulky, white, crystalline powder; odorless, or having a slight gaultheria-like odor, with a sweetish, afterward acid taste, and permanent in the air.

Soluble in 308 parts of water and in 2 parts of alcohol at 25°C . (77°F .); in 14 parts of boiling water and very soluble in boiling alcohol; also soluble in ether, absolute alcohol, and chloroform.

When heated to 156°C . (312.8°F .) the Acid begins to melt, and is completely melted at 157°C . (314.6°F .); at a higher temperature it is gradually dissipated without leaving more than 0.6 per cent. of fixed residue.

The saturated aqueous solution has an acid reaction upon blue litmus-paper, and is colored intensely bluish-violet (in high dilution violet-red) by ferric chloride T.S.

On adding to a small portion of Salicylic Acid, in a test-tube, about 1 Cc. of concentrated sulphuric acid, then, cautiously, about 1 Cc. of methyl alcohol in drops, and heating the mixture to boiling, methyl salicylate will be produced, which may be recognized by its odor.

On allowing a saturated alcoholic solution of the Acid to evaporate spontaneously in a glass or porcelain evaporating dish, in a place protected from dust, a perfectly white, crystalline residue should remain (absence of *iron*, *phenol*, or *coloring-matter*).

If 1 Gm. of the Acid be dissolved in an excess of cold sodium carbonate T.S., the liquid agitated with an equal volume of ether, and the ethereal solution allowed to evaporate spontaneously, the residue, if any, should be free from the odor of *phenol*.

On treating about 0.5 Gm. of Salicylic Acid, in a clean test-tube, with 10 Cc. of concentrated sulphuric acid, no color should be imparted to the latter within fifteen minutes (absence of *readily carbonizable organic impurities*).

Tests for Identity.—Given above.

Impurities.—Iron, phenol, coloring-matter, carbonizable matter. Details above. Hydrochloric acid. Details in Part V.

This body is *ortho-oxybenzoic acid*. It was discovered and named by Piria in 1839 as an oxidation product of salicin. It occurs natur-

ally in several of the volatile oils, notably that of wintergreen and sweet birch. The synthesis of salicylic acid has been accomplished in several ways. One is by fusing salicin with potassium hydroxide. It can also be made by treating orthocresol with potassa, but its commercial manufacture is by treatment of sodium carbolate with carbon dioxide, this being the celebrated *Kolbe's process*.

This can be best explained as the shoving of a molecule of CO_2 into a molecule of phenol. Phenol is $\text{C}_6\text{H}_5\text{OH}$, or it can be expressed as $\text{C}_6\text{H}_4\text{—}\frac{\text{OH}}{\text{H}}$; treat this with CO_2 , and we get salicylic acid, $\text{C}_6\text{H}_4\text{—}\frac{\text{OH}}{\text{COOH}}$. X 2
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It is interesting to note that in this case potassium carbolate cannot be used for the sodium carbolate. If the potassium carbolate is used, instead of getting ortho-oxybenzoic acid, we obtain the para-oxybenzoic acid.

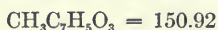
Salicylic acid occurs in fine white needles or light crystalline powder, soluble in 308 parts of water and 2 parts of alcohol, melting at 156° to 157°C . It gives a very characteristic and delicate reaction with ferric chloride, the latter agent giving a distinct red tint in an aqueous solution of one part salicylic acid to 400,000 parts water, or, roughly speaking, one grain dissolved in 7 gallons of water will give distinct reaction with ferric chloride. More concentrated solutions of salicylic acid or salicylates give a deep purple precipitate with ferric chloride.

Salicylic acid is a valuable antipyretic, antirheumatic, and antiseptic.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Several derivatives of salicylic acid are official: the salicylates of the metals sodium, lithium, ammonium, and strontium; of the alkaloïds, physostigmine and quinine; and the esters, methyl salicylate and phenyl salicylate.

METHYLIS SALICYLAS. Methyl Salicylate.



[METHYL SALICYLAS, PHARM. 1890]

An ester [$\text{C}_6\text{H}_4(\text{OH})\text{COOCH}_3$ 1:2], produced synthetically; it is the principal constituent of Oil of Gaultheria and Oil of Betula. For flavoring purposes, Oil of Gaultheria, Oil of Betula, and Methyl Salicylate may be regarded as identical products. It should be kept in well-stoppered bottles, protected from light.

A colorless liquid, having a characteristic, strongly aromatic, wintergreen odor, and a sweetish, warm, and aromatic taste.

Specific gravity: 1.180 to 1.185 at 25°C . (77°F .), according to the amount of moisture present.

Boiling-point: 219° to 221°C . (426.2° to 429.8°F .).

It is optically inactive.

Sparingly soluble in water; soluble, in all proportions, in alcohol, glacial acetic acid, and carbon disulphide.

The alcoholic solution is neutral or slightly acid to litmus-paper.

If a drop of Methyl Salicylate be shaken with a little water, and a drop of ferric chloride T.S. subsequently added, a deep violet color will be produced.

When heated on a water-bath, in a flask provided with a suitable condenser, it should yield no distillate having the characteristics of *alcohol* or *chloroform*.

If to 1 Cc. of Methyl Salicylate, contained in a capacious test-tube, 10 Cc. of potassium hydroxide T.S. be added, and the mixture agitated, a bulky, white, crystalline precipitate will be produced; then, if the test-tube, loosely corked, be allowed to stand in boiling water for about five minutes, with occasional agitation, the pre-

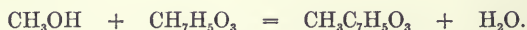
cipitate should dissolve, and form a clear, colorless, or faintly yellowish solution, without the separation of any oily drops, either on the surface or at the bottom of the liquid (absence of *volatile oils*, or of *petroleum*).

If the alkaline liquid thus obtained be subsequently diluted with about three times its volume of water, and a slight excess of hydrochloric acid added, a white, crystalline precipitate will be produced which, when collected on a filter, washed with a little water, and recrystallized from hot water, should respond to the tests of identity and purity given under *Acidum Salicylicum* (absence of *methyl benzoate*, etc.).

Tests for Identity.—Given above.

Impurities.—Alcohol, chloroform, other volatile oils, petroleum, methyl benzoate. Details above.

Remarks.—Methyl salicylate is made by treating methyl alcohol with salicylic acid in the presence of sulphuric acid, as shown by the following equation:



In this case, as in making all esters, the sulphuric acid acts as a dehydrating agent, removing the water formed during the reaction. Two natural forms of methyl salicylate are also recognized by the pharmacopœia, *oleum gaultheriæ* (oil of wintergreen), and *oleum betulæ* (oil of birch).

OLEUM GAULTHERIÆ. Oil of Gaultheria.

A volatile oil distilled from the leaves of *Gaultheria procumbens* Linné (Fam. *Ericaceæ*), rectified, if necessary, by steam distillation. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or almost colorless liquid, having a characteristic, strongly aromatic odor, and a sweetish, warm, and aromatic taste.

Specific gravity: 1.172 to 1.180 at 25° C. (77° F.).

Boiling-point: 218° to 221° C. (424.4° to 429.8° F.).

It is slightly levogyrate, up to —1° in a 100-Mm. tube, at 25° C. (77° F.).

In other respects it has the same properties as, and conforms to the reactions and tests given under, *Methylis Salicylas*.

OLEUM BETULÆ. Oil of Betula.

[OLEUM BETULÆ VOLATILE, PHARM. 1890]

A volatile oil obtained by maceration and distillation from the bark of the Sweet Birch, *Betula lenta* Linné (Fam. *Betulaceæ*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

It is optically inactive, but otherwise has essentially the same properties as, and conforms to the reactions and tests given under, *Oleum Gaultheriæ*.

Remarks.—The oil of sweet birch is recognized by the pharmacopœia because a large amount of the so-called *oil of wintergreen* is obtained by the distillation of the bark of birch. The differentiation between the oil of sweet birch and the oil of wintergreen is a matter of considerable difficulty. In fact, no less an authority than Professor Kremers claims that it is practically impossible to decide chemically whether a given sample is from wintergreen or from birch, although some therapeutists claim that the oil of birch is more irritating.

As methyl salicylate can be made by combining methyl hydroxide and salicylic acid, so oil of wintergreen and oil of birch can be split up into these two constituents by the process of treating the oil with

sodium hydroxide, when the methyl salicylate is decomposed by the following reaction:



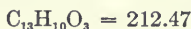
This process of breaking down an ester is called *saponification*, because the formation of soap from a fixed oil—an ester of glycerin—is accomplished by a practically identical process. (See p. 737.)

The saponification of the oil of wintergreen under the influence of sodium hydroxide is applied commercially in the manufacture of the so-called natural salicylate of sodium and salicylic acid. The process is a simple one, the oil being warmed in a porcelain dish on a water-bath, and then treated with molecular proportions of a solution of soda, when the sodium salicylate separates out in a white mass. On adding water the sodium salicylate dissolves, the solution is then filtered, and sodium salicylate obtained from the solution by granulation through evaporation. In preparing sodium salicylate by this process it is important that there be a slight excess of acid, otherwise the finished product will have an ugly gray color. Scrupulous pains must be taken to avoid any contact with iron during the manufacture, for the slightest trace of iron will color the sodium salicylate from pink to red. From this sodium salicylate a natural salicylic acid can be isolated by treatment with the requisite quantities of sulphuric acid and crystallization of the separated salicylic acid from alcohol.

These three forms of methyl salicylate are used as antirheumatics, but the less irritating sodium salicylate is usually preferable.

Dose.—Of all three, 1 Cc. (15 minims).

PHENYLIS SALICYLAS. Phenyl Salicylate.



[SALOL, PHARM. 1890]

The salicylic ester [$\text{C}_6\text{H}_4(\text{OH})\text{COOC}_6\text{H}_5$ 1:2] of phenyl.

A white, crystalline powder, having a faint, aromatic odor, and a slight, but characteristic taste.

Soluble in 2333 parts of water and in 5 parts of alcohol at 25° C. (77° F.); very soluble in hot alcohol, and in ether, chloroform, and fixed and volatile oils.

When heated to 42° C. (107.6° F.) it melts, and at a higher temperature is consumed, leaving no weighable residue.

If to the alcoholic solution be added diluted ferric chloride T.S., a violet color is produced.

If 0.2 to 0.3 Gm. of Phenyl Salicylate be dissolved in a little warm sodium hydroxide T.S., and the solution be then acidified with hydrochloric acid, salicylic acid separates, and the odor of phenol is recognizable.

Phenyl Salicylate should not redden moistened blue litmus-paper (absence of free acids).

If 1 Gm. of Phenyl Salicylate be shaken with 50 Cc. of water, the liquid filtered, and 5 drops of ferric chloride T.S., previously diluted with 20 volumes of water, be added, the filtrate should show either no color, or at most a trace (limit of *uncombined phenol* or *salicylic acid*).

Tests for Identity.—Given above.

Impurities.—Free acid, uncombined phenol, and salicylic acid. Details above. Sulphates and chlorides. Details in Part V.

This graphic formula, $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{OH} \\ \text{COOC}_6\text{H}_5 \end{smallmatrix}$, shows that salol is salicylic

acid with the carboxyl hydrogen replaced by the phenyl group (C_6H_5). It can also be written as phenyl salicylate, $C_6H_5C_7H_5O_3$.

Salol is an artificial ester obtained by the treatment of phenol with salicylic acid in the presence of phosgen, sulphuric acid not being indicated in this case, since its action would be too violent. The equation of manufacture is—



Salol is a remedy of comparatively recent origin, and has been found admirable in rheumatic cases, it having a less irritating action than have the other salicylates.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Salol is interesting because insoluble in acids, but freely soluble in alkalis. On account of this peculiarity it is used for a coating for enteric pills, such pills being desired to pass through the acid juices of the stomach undissolved, but intended to dissolve in the alkaline juices of the intestines (p. 321).

So successful has salol proved that a large number of derivatives from it are now found on the market.

Salophene is acetyl-para-amido-salol, $p-C_6H_4 \begin{smallmatrix} \nearrow OCOC_6H_4OH \\ \nwarrow NHCOCH_3 \end{smallmatrix}$.

The structure of this body can be best explained by the stages of its manufacture.

Salol is converted into para-nitro-salol, $p-C_6H_4 \begin{smallmatrix} \nearrow OCO-C_6H_4OH \\ \nwarrow NO_2 \end{smallmatrix}$. This, by reduction, gives para-amido-salol, $p-C_6H_4 \begin{smallmatrix} \nearrow OCO-C_6H_4OH \\ \nwarrow NH_2 \end{smallmatrix}$, and this, treated with acetic acid, yields salophene.

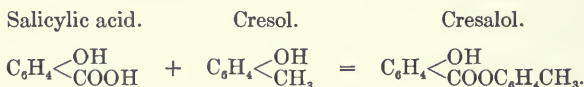
Salophene is used as a salol substitute in rheumatic and gastric troubles, it being considered safer than salol.

Saliphenic, $C_6H_4 \begin{smallmatrix} \nearrow OC_2H_5 \\ \nwarrow NHC_6H_5O_2 \end{smallmatrix}$.—This body is a salicyl-phenetidin, and is mentioned in passing merely to call attention to the confusing similarity of the names of these two substances.

Salicylamide, $C_6H_4 \begin{smallmatrix} \nearrow CONH_2 \\ \nwarrow OH \end{smallmatrix}$.—This is made by treating oil of wintergreen with ammonia gas, and is used as a salicylic acid substitute.

Cresalol, $C_6H_4 \begin{smallmatrix} \nearrow OH \\ \nwarrow COOC_6H_4CH_3 \end{smallmatrix}$.—Salol is salicylic acid combined with the phenol, C_6H_4OH . Cresalol is salicylic acid combined with cresol, $C_6H_4 \begin{smallmatrix} \nearrow CH_3 \\ \nwarrow OH \end{smallmatrix}$.

It is made by combining sodium salicylate and sodium cresylate in the presence of phosphorous oxychloride, $POCl_3$. The reaction can best be shown by the two compounds uncombined with sodium, viz.:



DIOXYBENZOIC ACIDS

We have already seen that salicylic acid is a mono-oxybenzoic acid, or a phenol in which one atom of hydrogen has been replaced by the carboxyl group. In the same way the dioxyphenols, by substitution of one atom of hydrogen with carboxyl, yield dioxybenzoic acids.

Thus, pyrocatechin, $\text{o-C}_6\text{H}_4(\text{OH})_2$, yields protocatechuic acid, $\text{C}_6\text{H}_3\text{COOH}$ $\begin{smallmatrix} (\text{OH})_2 (3: 4) \\ (1) \end{smallmatrix}$.
 Resorcin, $\text{m-C}_6\text{H}_4(\text{OH})_2$, yields resorecylic acid, $\text{C}_6\text{H}_3\text{COOH}$ $\begin{smallmatrix} (\text{OH})_2 (3: 5) \\ (1) \end{smallmatrix}$.
 Hydroquinone, $\text{p-C}_6\text{H}_4(\text{OH})_2$, yields gentisinic acid, $\text{C}_6\text{H}_3\text{COOH}$ $\begin{smallmatrix} (\text{OH})_2 (2: 5) \\ (1) \end{smallmatrix}$.

These acids are of little importance in pharmacy.

Gentisinic acid is a decomposition product of gentisin (p. 712), and protocatechuic acid is of interest only by reason of an important derivative of its aldehyde.

Protocatechuic aldehyde has the formula $\text{C}_6\text{H}_3\text{CHO}$ $\begin{smallmatrix} \text{CHO} \\ \text{OH} \\ \text{OH} \end{smallmatrix}$, and the methyl ether of this aldehyde is vanillin.

VANILLINUM. Vanillin.



Methylprotocatechuic aldehyde [$\text{C}_6\text{H}_3\text{OH.OCH}_3\text{.COH}$ 4:3:1], occurring naturally in vanilla, or made artificially from several orthodihydroxybenzene derivatives.

Fine, white, crystalline needles, having the odor and taste of vanilla, and having an acid reaction.

Soluble in about 100 parts of water at 25°C . (77°F .) and in 15 parts at 80°C . (176°F .) It is easily soluble in alcohol, ether, glycerin, or chloroform.

When heated to between 80° and 81°C . (176° and 177.8°F .) it melts, and at 285°C . (545°F .) it can be distilled without decomposition in a current of carbon dioxide, leaving no residue.

It is easily soluble in aqueous solutions of alkali hydroxides, and from the combinations thus formed it is precipitated at once by the addition of acids.

An aqueous solution of Vanillin gives, with ferric chloride T.S., a blue color; if the mixture be boiled, the blue color changes to brown, and on cooling a white precipitate of dihydro-divanillin separates.

Vanillin is extracted completely from its solution in ether by shaking with a saturated aqueous solution of sodium bisulphite, from which solution it is precipitated by the addition of sulphuric acid.

An aqueous solution of Vanillin will give, on the addition of lead acetate T.S., a white precipitate of a lead compound of vanillin, soluble in hot water, and crystallizing, on cooling, in scales.

On warming 0.1 Gm. of Vanillin with concentrated alcoholic solution of sodium hydroxide, adding chloroform and again warming, it should not give an odor of phenyl-isocyanide (absence of *acetanilide*).

Tests for Identity.—Given above.

Impurities.—Acetanilide, given above.

Remarks.—Vanillin is the odorous principles of the vanilla bean, which has long been popular as a flavoring agent. The extraction of vanilla from vanilla beans is a matter of comparatively little difficulty, but the small yields caused its commercial extraction to be at a cost almost prohibitive. The researches of Tiemann, however, proved the structural formula of the substance, and when this was known, the investigator found it a matter of no great difficulty to prepare vanillin from the cheaper sources than from vanilla beans. The first vanillin made synthetically was made from coniferin found in the cambial juices of some species of pine. Vanillin from this source was prepared by Tiemann and Haarmann in their chemical factory at Holzminden, and soon became an article of commerce. A few years later, however, Tiemann discovered a simpler and more economic source of the production of vanillin in eugenol, a constituent

of the oil of cloves, from which it is obtained by oxidation with potassium permanganate.

Vanillin occurs in the form of white crystals, melting at 81° C., freely soluble in alcohol, and much less so in water. It possesses a delightful odor of the vanilla beans, and is now very largely used instead of vanilla beans, both in flavoring extracts and particularly in perfumery.

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

VANILLA. *Vanilla*.

The cured, full grown, but immature fruit of *Vanilla planifolia* Andrews (Fam. *Orchidaceæ*).

Constituents.—Vanillin, volatile oil, fixed oil, resin, sugar, mucilage, etc.

Remarks—There are a great variation in quality of commerce vanilla beans, ranging from Mexican beans, the finest, to the Tahiti bean, the poorest—so poor that they should not be used. The poorer the quality, the more mucilage and less aroma the bean possesses.

Medical Properties.—Carminative and aphrodisiac. Chiefly used, however, as a flavor. For this purpose the official tincture (p. 237) is employed.

Dose.—1 Gm. (15 grains).

CHAPTER XLV

GALLIC AND TANNIC ACIDS

ASTRINGENT DRUGS

ACIDUM GALLICUM. Gallic Acid.



AN organic acid [$\text{C}_6\text{H}_3(\text{OH})_3\text{COOH} + \text{H}_2\text{O}$], usually prepared from tannic acid.

White, or pale fawn-colored, silky, interlaced needles, or triclinic prisms; odorless; having an astringent and slightly acidulous taste; permanent in the air.

Soluble in 83.7 parts of water and in 4.14 parts of alcohol at 25° C. (77° F.); in 3 parts of boiling water and in 1 part of boiling alcohol; also soluble in 40 parts of ether and in 12 parts of glycerin; very slightly soluble in chloroform, benzene, or petroleum benzin.

When heated at 100° C. (212° F.) the Acid loses its water of crystallization (nearly 9.58 per cent.). At about 200° C. (392° F.) it begins to melt, and at a higher temperature it is gradually decomposed. At a low red heat it is consumed without leaving a residue.

An aqueous solution of Gallic Acid has an acid reaction upon blue litmus-paper.

If 5 Cc. of a cold saturated aqueous solution of the Acid be treated, in a watch-glass, with 6 drops of sodium hydroxide T.S., the liquid will gradually acquire a deep green color, which is changed to reddish or brownish-red by acids.

Gallic Acid neither colors nor precipitates pure ferrous salts, but forms a bluish-black precipitate with ferrie salts.

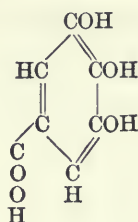
On adding to a cold saturated aqueous solution of Gallic Acid some calcium hydroxide T.S., a bluish-white precipitate will form, where the test solution is temporarily in excess, and will disappear on shaking. When the test solution has been added in excess, the precipitate no longer dissolves, and the liquid acquires a tint which is blue by reflected and green by transmitted light, and becomes pink on the addition of a large excess of calcium hydroxide T.S. (distinction from *tannic acid*).

An aqueous solution of the Acid should not precipitate alkaloids, gelatin T.S., albumin T.S., or starch T.S. (difference from and absence of *tannic acid*).

Tests for Identity.—Given above.

Impurities.—Tannic acid. Details above.

Remarks.—The graphic formula of gallic acid is—



$\text{C}_6\text{H}_2(\text{OH})_3\text{COOH}$, or, massed together, $\text{HC}_7\text{H}_5\text{O}_5$.

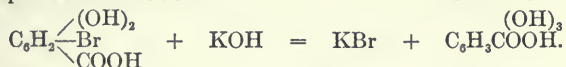
Such is gallic acid, which is benzene with three hydroxyls and one carboxyl replacing four hydrogen atoms.

While protocatechuic acid represents dioxybenzoic acid, it will be seen from the above formula that gallic acid is a *trioxybenzoic acid*. Gallic acid was isolated from nutgall by Scheele in 1785, who gave it the name from the source of its supply.

Gallic acid can be made synthetically by fusing of bromoprotocatechuic acid with potassa, as shown in the following equation:

Bromoprotocatechuic acid.

Gallic acid.

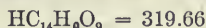


It is usually obtained by macerating nutgalls with water long enough (several weeks) to hydrolyze the tannin into gallic acid. The cold acid aqueous liquid, in which but little gallic acid and much of the foreign matter dissolves, is expressed and thrown away. The press cake is then treated with boiling water, in which the gallic acid freely dissolves, and the hot infusion set aside to permit crystallization of the acid.

Gallic acid is a valuable astringent, of particular service as a harmless agent for stopping minor hemorrhages, such as nosebleed. In such case snuffing a small quantity of gallic acid up the nose usually staunches the flow of blood. It is given as an intestinal astringent, and is also used externally; for this purpose the official ointment of gallic acid is recommended.

Dose.—1 Gm. (15 grains).

Bismuth subgallate is now official, and has been described among the preparations of bismuth (p. 596).

ACIDUM TANNICUM. Tannic Acid.

A monobasic organic acid [$\text{C}_{13}\text{H}_9\text{O}_7\text{COOH}$], obtained from nutgall.

A light yellowish, amorphous powder, gradually turning darker when exposed to air and light, usually cohering in the form of glistening scales or spongy masses, odorless, or having a faint, characteristic odor, and a strongly astringent taste.

Soluble in about 0.34 part of water and in about 0.23 part of alcohol at 25°C . (77°F .); very soluble in boiling water and in boiling alcohol; also in about 1 part of glycerin, with the application of a moderate heat; freely soluble in diluted alcohol, sparingly in absolute alcohol; almost insoluble in absolute ether, chloroform, benzene, or petroleum benzin.

When heated on platinum foil, the Acid is gradually consumed, leaving not more than 0.2 per cent. of ash.

An aqueous solution of Tannic Acid reddens blue litmus-paper.

The addition of a small quantity of ferric chloride T.S. to an aqueous solution of the Acid produces a bluish-black color or precipitate.

On adding to an aqueous solution (1 in 100) of Tannic Acid a small quantity of calcium hydroxide T.S., a pale bluish-white flocculent precipitate is produced which is not dissolved on shaking (difference from *gallic acid*), and which becomes more copious and of a deeper blue by the addition of a moderate excess of calcium hydroxide T.S., while a large excess of the latter imparts a pale pinkish tint to the solution.

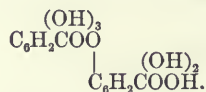
The aqueous solution of the Acid produces precipitates with most alkaloids and glucosides, and with test solutions of gelatin, albumin, and starch (distinction from *gallic acid*).

If 2 Gm. of Tannic Acid be dissolved in 10 Cc. of boiling water, and the liquid allowed to cool, no turbidity should be produced on diluting 5 Cc. of the solution with 10 Cc. of alcohol (absence of *gum* or *dextrin*) or with 10 Cc. of water (absence of *resinous substances*).

Tests for Identity.—Given above.

Impurities.—Gallic acid, gum, dextrin, resinous substances. Details above.

Remarks.—The graphic formula of tannic acid (or *tannin*, as it is usually called) is—



In other words, it is digallic acid.

Gallic acid is $\text{C}_7\text{H}_6\text{O}_5$; two molecules of this equals..... $\text{C}_{14}\text{H}_{12}\text{O}_{10}$

Subtract from this one molecule of water..... H_2O

We have left one molecule of tannic acid..... $\text{C}_{14}\text{H}_{10}\text{O}_9$

As will be seen from the above, tannic acid represents gallic acid deprived of a molecule of water; this water is removed by treatment of gallic acid with concentrated sulphuric acid. On the other hand, tannic acid can be converted into gallic acid by heating with diluted sulphuric acid.

The tannic acid of the pharmacopœia is but one of the very interesting series of compounds that we call the tannins (or tannoids). Most of these, instead of being pure digallic acid, are glucosides thereof. A very large number of plants yield various forms of tannins, and very few of these are identical.

Efforts have been made toward the classification of the tannins, and the first groups into which they are divided are according as to whether they form with iron salts a green black precipitate or a blue-black precipitate. The most modern classification of the tannins is that of Kunz Krause.

NON-GLUCOSIDAL TANNIDS: Such as gallic acid; protocatechuic acid, digallic acid, etc.

GLUCOSIDAL TANNIDS:

(a) *Glycotannoids.*

Protocatechuic acid derivatives, such as cinchotannic acid, caffetannic acid, etc. Gallic acid derivatives, such as pomegranate root tannin.

(b) *Phloroglucin tannoids.*

Protocatechuic acid derivatives, such as quebracho tannic acid, catechutannic acid, kolatannin, etc.

Gallic acid derivatives, none yet known.

The protocatechuic acid derivatives mentioned above include those tannoids turning green black with ferric chloride; while the "gallic acid derivatives" turn blue black with ferric chloride.

It will be noticed in the pharmacopœial description that tannin is precipitated by gelatin (as discussed under Clarification, see p. 136) as a leathery substance, leather representing this very substance. Fresh animal hide consists of gelatin combined with a mass of animal fibers, and when this is macerated in a solution of tannin (generally from oak bark), it is converted into leather or the tannate of gelatin. The deep blue-black precipitate produced by the treatment of tannin with ferric chloride solution is the basis of most of our writing ink, which is made by treating nutgall infusion with iron salts and adding some gummy substance which will hold the precipitate in suspension.

Tannin is obtained by exposing powdered nutgalls to a damp atmosphere for several days, and then macerating with a small quantity of ether, and expressing the liquid, repeating the maceration and expression with ether containing one-sixteenth its bulk of water. The reason of this process of extraction is puzzling, since the tannin so obtained is practically insoluble in strong ether. Tannin can also be extracted from nutgall with a mixture of alcohol and ether, and in a patented process acetic ether is the solvent employed.

Tannic acid can also be made as above mentioned by treatment of gallic acid with diluted sulphuric acid.

Tannic acid is one of the most valuable astringents, and is used both internally and externally. When used internally, it is given for diarrhea. For this purpose, however, it is usually advisable to administer a tannin-bearing drug, such as catechu or krameria.

Externally, it is used to staunch the flow of blood, and for gargles in sore throat and as an application for piles.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

The official preparations of tannin are glycerite of tannic acid, troches of tannic acid, and ointment of tannic acid.

DRUGS CONTAINING ASTRINGENT PRINCIPLES

KRAMERIA. *Krameria*. *Rhatany*.

The dried root of *Krameria triandra* Ruiz and Pavon (Peruvian *Krameria*), *Krameria Ixina* Linné (Savanilla *Krameria*), or of *Krameria argentea* Martius (Para or Brazilian *Krameria*) (Fam. *Krameriaceæ*).

Constituents.—Kramerotannic acid, which consists of pure tannin, phlobaphene, and extract; krameric acid; an alkaloid having formula $C_{10}H_{13}NO_3$; red coloring-matter, starch, etc.

Medical Properties.—Astringent, used in diarrhea as *fluidextract* (p. 247), *tincture* (p. 229), and *syrup* (p. 196). Also used for the throat in the form of the official lozenges (p. 332).

Dose.—1 Gm. (15 grains).

GERANIUM. Geranium. Cranesbill.

The dried rhizome of *Geranium maculatum* Linné (Fam. *Geraniaceæ*).

Constituents.—Tannin, gallic acid, mucilage, resin, etc.

Medical Properties.—Astringent. The *fluidextract* (p. 248) is official.

Dose.—1 Gm. (15 grains).

RUBUS. Rubus. Blackberry Root.

The dried bark of the rhizome of *Rubus villosus* Aiton, *Rubus nigrobaccus* Bailey, or of *Rubus cuneifolius* Pursh (Fam. *Rosaceæ*).

Constituents.—Tannin, *villosin* (bitter glucoside), etc.

Medical Properties.—Astringent. Used in diarrhea as the official *fluidextract* (p. 247) or *syrup* (p. 199).

Dose.—1 Gm. (15 grains).

HÆMATOXYLON. Hematoxylon. Logwood.

The heart-wood of *Hæmatoxylon campechianum* Linné (Fam. *Leguminosæ*).

Constituents.—Tannin, *hematoxylin*, a neutral principle having the formula $C_{16}H_{14}O_6$, and giving the wood its color.

Remarks.—The “heart wood” means the heavier older wood found in the interior of a trunk. Its technical name is the “duramen,” while the lighter, younger, outer wood is called the “alburnum.” The hematoxylin mentioned above has chemical value as an indicator in volumetric analysis (p. 988).

Chief use of logwood is in dyeing, a decoction of the wood mixed with copperas coloring fabrics a deep black. For dyeing purposes, however, the wood is generally submitted to a fermentation process, when it assumes a green, metallic lustre. Such wood should not be used in medicine.

Medical Properties.—Astringent, when used medicinally, and then administered as the official extract (p. 284).

HAMAMELIDIS CORTEX. Hamamelis Bark. Witch Hazel Bark.

The bark and twigs of *Hamamelis virginiana* Linné (Fam. *Hamamelidaceæ*).

Constituents.—Tannin, bitter principle, chlorophyll, trace of volatile oil.

Remarks.—This new official has been introduced because used in making distilled extract of witch hazel. (See *Aqua Hamamelidis*, U. S. P., p. 174.)

Medical Properties.—Astringent of little value.

Dose.—2 Gm. (30 grains).

QUERCUS. White Oak.

[QUERCUS ALBA, PHARM. 1890]

The dried bark of *Quercus alba* Linné (Fam. *Cupuliferae*), collected from trunks or branches ten to twenty-five years of age, and deprived of the periderm.

Constituents.—Tannin (quercitannic acid), which is not a glucoside; oak red, pectin, etc.

Remarks.—The word “periderm,” in the official definition, means the outer corky layer of the bark.

Medical Properties.—Astringent. Used largely in the tanning of leather.

Dose.—1 Gm. (15 grains).

HAMAMELIDIS FOLIA. Hamamelis Leaves.

[HAMAMELIS, PHARM. 1890]

The dried leaves of *Hamamelis virginiana* Linné (Fam. *Hamamelidaceae*), collected in autumn.

Constituents.—Tannin, trace of volatile oil.

Medical Properties.—Astringent. Of even less value than the bark. The fluidextract is official (p. 248).

Dose.—2 Gm. (30 grains).

ROSA GALLICA. Red Rose.

The dried petals of *Rosa gallica* Linné (Fam. *Rosaceae*), collected before expanding.

Constituents.—Tannin, gallic acid, trace of volatile oil, quercetin, color, etc.

Medical Properties.—Astringent tonic. Rose Honey, U. S. P. (p. 203). Formerly highly esteemed as a wash for sore mouth. Syrup of Rose, U. S. P. (p. 199), used as a flavor and as a mild astringent. Both of these are made from the official fluidextract (p. 248).

Pale rose, a former official drug, was dropped in the last revision. It contains only a trace of tannin, and was used chiefly as a flavor. Bear in mind that while the official *stronger rose water* (p. 175) is made from these two roses, the official *oil of rose* comes from a different species of rose. (See p. 820.)

GALLA. Nutgall.

An excrescence on *Quercus infectoria* Olivier (Fam. *Cupuliferae*), caused by the punctures and deposited ova of *Cynips tinctoria* Olivier.

Constituents.—Tannin, 50 per cent. (a glucosidal form that hydrolyzes to glucose and digallic acid); gallic acid, 3 per cent.; pectose; a ferment (pectase), etc.

Remarks.—The above official definition (“an excrescence on *Quercus infectoria*”) should be analyzed if it is to be clearly understood. Excrescence means, literally, “an outward growth.” In medicine, a boil, a mole, and a wart are, alike, excrescences, likewise, to a certain extent, the swellings caused by insect-bites, all such “outward growths” being the result of abnormal development of tissue caused by some irritation. So, in plainer English, we may say that nutgall

is a swelling on *Quercus infectoria*, a swelling "caused by punctures and deposited ova of *Cynips tinctoria*." To explain that part of the definition just quoted is easy. It has just been said that when an insect bites us, an "excrescence" is formed on the injured part. Likewise, official nutgall, the excrescence on a special variety of oak, is caused by the bite (puncture) of a special insect—*Cynips tinctoria*. This insect not only stings the oak, but also deposits in the puncture its eggs (*ova*, plural for *ovum*, an egg), and these are the foreign bodies that increase the irritation.

To make a résumé, the insect, *Cynips tinctoria*, with that unerring instinct given by Providence, chooses the tissue of an oak tree as the best place in which its young may be hatched, and, accordingly, pierces the younger branches and deposits its eggs in the hole.

The stung branch swells just as does a stung arm, and still more so, by reason of the irritating particles (the eggs) contained in the wound. So the swelling continues, until a round knob, about the size of a cherry, is formed—a morbid growth not unlike a wen or a tumor on human beings. While the excrescence, the nutgall, is growing, the insect eggs are undergoing that process of development called hatching, and when the nutgall assumes its full size, the eggs will have hatched and the young and hungry grubs enter into their curious world. All insects have two stages of existence, as seen, for instance, in the caterpillar, the larval stage, and the butterfly, the most highly developed stage of the same animal. In their larval state insects are destitute of wings, and in just such condition is *Cynips tinctoria* as it emerges from its egg within the nutgall. To its voracious appetite the soft tissue of the nutgall is exactly suited, and within this dark home it lives its larval life, and only after it has undergone the second transformation and becomes an insect does it desire to see the outer world. Then it bores its way through the walls of its prison cell, entering the living world about six months after the egg was laid.

As a matter of fact, nutgall should be collected before the insect has escaped, hence the gall should not be punctured, and on slicing should show a cavity containing remnants of the insect.

As already mentioned (p. 789), nutgall is the source of both official tannin and of gallic acid.

Medical Properties.—Valuable astringent. The official tincture (p. 227) is used in diarrhea mixtures, while the official ointment (p. 346) is largely used for hemorrhoids.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

GAMBIR. Gambir.

[TO REPLACE CATECHU, PHARM. 1890]

An extract prepared from the leaves and twigs of *Ourouparia Gambir* (Hunter) Baillon (Fam. *Rubiaceæ*).

Constituents.—Catechu-tannic acid; catechin, etc.

Remarks.—This new official replaces the formerly official catechu. It occurs in pale brownish-gray or light brown cubes, quite different in appearance from the dark brown catechu. It is used extensively as a dye-stuff, coloring black when combined with iron salts.

Medical Properties.—Astringent. Administered in diarrhea mixtures as the compound tincture (p. 227), and for throat troubles as the official lozenges (p. 332).

Dose.—1 Gm. (15 grains).

Catechu, the extract of the wood of *Acacia catechu*, was official until the last revision, when it was replaced by gambir, with which it is identical as far as constituents and medical properties are concerned.

KINO. Kino.

The inspissated juice of *Pterocarpus Marsupium* Roxburgh (Fam. *Leguminosæ*).

Constituents.—Kinotannic acid; kinoic acid; kinoin, $C_{14}H_{12}O_6$; kino red, $C_{28}H_{22}O_{11}$, etc.

Remarks.—The only point worth noting regarding kino is the fact, already mentioned (p. 229), that kino contains an enzyme which causes the tincture to gelatinize, unless the recipe of U. S. P. VIII. is strictly followed.

Medical Properties.—Astringent. Administered in diarrhea mixtures as tincture (p. 229).

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

CHAPTER XLVI

AROMATIC HYDROCARBONS CONTAINING EIGHT, NINE, AND TEN CARBON ATOMS

TERPENES AND THEIR DERIVATIVES

THE hydrocarbons of the series C_2H_{2n-6} , containing eight carbon atoms, have the empiric formula,

$$\begin{array}{l} C_8H_{10} \\ C_n \\ H_{2n-6} \end{array} = \begin{array}{l} C_8 \\ \\ H_{2 \times 8} \end{array} = 16. \quad 16 - 6 = 10. \quad \text{Hence } H_{10}.$$

This formula includes the three forms of dimethyl-benzene, $C_6H_4-(CH_3)_2$, that is, ortho-, meta-, and para-xylenes, and also ethyl benzene, $C_6H_5C_2H_5$. From the three xylenes are derived the three forms of *phthalic acid*, $C_6H_4(COOH)_2$, which are mentioned in passing because the anhydride of the ortho acid in condensation with phenol yields *phenolphthalein*—so largely used as an indicator in volumetric analysis.

Phenolphthalein is now enjoying considerable vogue as a purgative, being the main constituent of such proprietaries as *probilin*, *phenolax*, etc.

As a derivative of ethyl benzene might be cited *epinephrin*, $C_6H_3(OH)_2CHOH-CH_2NHCH_3$, obtained from the suprarenal glands (p. 940), and frequently prescribed under the trade names, *adrenalin*, *adrin*, *adnephryn*, etc.

Among the hydrocarbons of this series containing nine carbon atoms are the three trimethyl benzenes, $C_6H_3(CH_3)_3$, of which the most important is the symmetric form, *mesitylene*; the three methyl-

ethyl benzenes, $\text{C}_6\text{H}_4\text{CH}_3$; and the two propyl benzenes, $\text{C}_6\text{H}_5\text{C}_3\text{H}_7$, of which the most important is the isopropyl form, *cumene*, $\text{C}_6\text{H}_5\text{CH}(\text{CH}_3)_2$.

Of far greater importance in pharmacy are the unsaturated hydrocarbons of this series, *allyl benzene*, $\text{C}_6\text{H}_5\text{CH}=\text{CH}-\text{CH}_3$, and *iso-allyl-benzene*, $\text{C}_6\text{H}_5\text{CH}_2\text{CH}=\text{CH}_2$. Neither of these bodies in themselves are of pharmaceutical interest, but each furnishes a long line of derivatives, many of which are constituents of volatile oils. Thus the alcohol of allyl-benzene is *cinnamic alcohol*, $\text{C}_6\text{H}_5\text{CH}=\text{CHCH}_2\text{OH}$, and this on oxidation yields *cinnamic aldehyde*, $\text{C}_6\text{H}_5\text{CH}=\text{CHCHO}$, and *cinnamic acid*, $\text{C}_6\text{H}_5\text{CH}=\text{CHCOOH}$.

The following derivatives of allyl benzene and iso-allyl-benzene are of pharmaceutical importance:

Cinnamic alcohol.

Cinnamic aldehyde, U. S. P.

Cinnamic acid.

Coumaric acid.

Coumarin.

Anethol.

Eugenol, U. S. P.

Safrol, U. S. P.

Apiol.

CINNALDEHYDUM. Cinnamic Aldehyde.

$\text{C}_9\text{H}_8\text{O} = 131.07$

An aldehyde obtained from Oil of Cinnamon or prepared synthetically, containing not less than 95 per cent. of pure Cinnamic Aldehyde [$\text{C}_6\text{H}_5\text{CH}=\text{CH}\cdot\text{COH}$]. It is nearly identical with the oil distilled from Cassia Cinnamon, and should be kept in small, amber-colored, well-stoppered bottles.

A colorless liquid, having a cinnamon-like odor and a burning, aromatic taste.

Specific gravity: about 1.047 at 25° C. (77° F.). Optically inactive.

It boils at about 250° C. (482° F.), with partial decomposition.

Cinnamic Aldehyde should solidify when the temperature is reduced by a freezing mixture of ice and salt, and should melt again at -7.5° C. (18.5° F.).

It is sparingly soluble in water, soluble in all proportions in alcohol, ether, and fixed and volatile oils.

Tests for Identity.—Given above.

Impurities.—Chlorinated products. Test, the copper wire method given under Benzaldehyde (p. 776).

Assay.—Details in Part V.

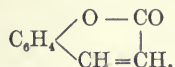
Remarks.—This synthetic volatile oil is practically identical with oil of cinnamon, which will be discussed in the next chapter. When used medicinally, it is employed as a carminative.

Dose.—0.05 Cc. (1 minim).

Both cinnamic aldehyde and cinnamic acid are found in nature elsewhere than in oil of cinnamon; that class of drugs called *balsams* being oleoresins containing benzoic and cinnamic acids, and usually some traces of the corresponding aldehydes.

Cinnamic acid is a crystalline solid of agreeable aromatic odor, and can be converted into oxycinnamic acid (*coumaric acid*), $\text{C}_6\text{H}_4\text{CH}(\text{OH})=\text{CHCOOH}$. When

coumaric acid is treated with acetic acid anhydride a molecule of water separates, with the formation of *coumarin*, the formula of which is—



Coumarin was so named because first obtained from the Tonka bean, the fruit of *Diptyryx odorata* (formerly called *Coumarouna odorata*).

The synthesis of coumarin from coumaric acid was accomplished by Tiemann in 1877, and at present almost all the coumarin in commerce is made artificially.

Coumarin is also found in the aromatic herbs, such as deer's tongue (*Liatris odorissima*) and mellilot (*Mellilotus alba*). There are also traces in the ordinary grasses, in which it becomes prominent on drying; producing the odor of new-mown hay.

Coumarin occurs in white crystals, melting at 67° C. (152.3° F.), soluble in 14 parts of alcohol, and sparingly soluble in water. It is used for flavoring the cheaper grade of vanilla extracts (see Compound Tincture of Vanillin of the National Formulary).

Among other derivatives of allyl-benzene we find: *Ferulaic acid* (methyl-oxy-cinnamic acid), $\text{C}_6\text{H}_5 \begin{cases} \text{OH} \\ \text{OCH}_3 \\ \text{CH} = \text{CHCOOH} \end{cases}$ the chief constituent of the resin of asa-fetida (p. 856).

Anethol (para-propenyl-anisol), formula, $\text{C}_6\text{H}_4 \begin{cases} \text{OH} \\ \text{CH} = \text{CHCH}_3 \end{cases}$.

This body is the methyl ether of the phenol of allyl-benzene, $\text{C}_6\text{H}_5 \begin{cases} \text{OH} \\ \text{CH} = \text{CH} - \text{CH}_3 \end{cases}$, and is the chief constituent of the oils of anise, star anise, and fennel. It is a crystalline solid, melting at 21° C. (69.4° F.).

Two derivatives of iso-allyl-benzene ($\text{C}_6\text{H}_5\text{CH}_2\text{CH}=\text{CH}_2$) are worthy of notice.

EUGENOL. Eugenol.

$$\text{C}_{10}\text{H}_{12}\text{O}_2 = 162.86$$

An unsaturated, aromatic phenol [$\text{C}_6\text{H}_3(\text{OH})(\text{OCH}_3).\text{C}_3\text{H}_5$, 4:3:1], obtained from Oil of Cloves and other sources. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow, thin liquid, having a strongly aromatic odor of cloves, and a pungent and spicy taste. Exposure to air causes it to become darker and thicker.

Specific gravity: 1.072 to 1.074 at 25° C. (77° F.).

Eugenol is miscible with alcohol in all proportions, and should be soluble in 2 parts of 70 per cent. alcohol.

Boiling-point: 251° to 253° C. (483.8° to 487.4° F.).

It is optically inactive.

When 1 part of Eugenol is dissolved in 12 parts of sodium hydroxide T.S. and 18 parts of water are added, a clear solution should result, which becomes turbid when exposed to the air.

A mixture of 1 part of Eugenol and 20 parts of hot water should redden litmus-paper very slightly. Five Cc. of the cold, clear filtrate from this mixture, upon the addition of 1 drop of ferric chloride T.S., should show a transient grayish-green color, but not a blue or violet color (absence of *phenol*).

Tests for Identity.—Given above.

Impurity.—Phenol. Details above.

Remarks.—Eugenol is allyl guaiacol, $\text{C}_6\text{H}_3 \begin{cases} \text{CH}_2\text{CH} = \text{CH}_2 \text{ (1)} \\ \text{OCH}_3 \text{ (3)} \\ \text{OH} \text{ (4)} \end{cases}$, or

the methyl ether of the divalent phenol from iso-allyl-benzene, $\text{C}_6\text{H}_3 \begin{cases} \text{CH}_2\text{CH} = \text{CH}_2 \\ \text{OH} \\ \text{OH} \end{cases}$, and is the chief constituent of the oil of cloves.

It is an aromatic oily liquid, boiling at 247° C. (476.3 F.).

Its conversion into vanillin has been already described (p. 787). Its medical properties are similar to oil of cloves, from which it is derived.

Dose.—0.2 Cc. (3 minims).

SAFROLUM. Safrol.



The methylene ether of allyl pyrocatechol [$C_6H_3.C_3H_5.(OOCH_2) 1:3:4$] found in oil of sassafras, camphor oil, and other volatile oils, purified, if necessary, by repeated chilling and crystallization.

A colorless or faintly yellow liquid with a sassafras-like odor.

Specific gravity: 1.105 to 1.106 at 25° C. (77° F.).

Boiling-point: about 233° C. (451.4° F.).

It is optically inactive. On cooling to -20° C. (-4° F.) or below, it solidifies to a mass of crystals, which do not melt below 11° C. (51.8° F.).

Soluble in about an equal volume of strong alcohol and in about 30 parts of 70 per cent. alcohol. Miscible in all proportions with ether and chloroform.

Tests for Identity.—Given above.

Safrol is allyl pyrocatechin-methylen-ether, $C_6H_3O_2 \begin{matrix} CH_2-CH=CH_2 (1) \\ O > CH_2 (3) \end{matrix}$ (4),

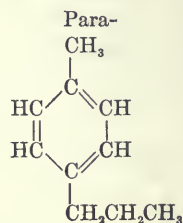
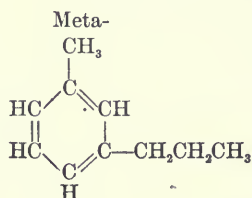
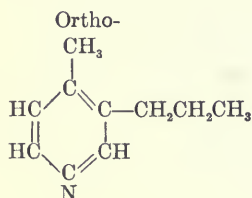
and represents eugenol, with its methyl group replaced by the methylene group ($\begin{matrix} -O \\ -O > CH_2 \end{matrix}$). Safrol is the chief constituent of oil of sassafras, and is a crystalline solid, melting at 8.5° C. and boiling at 233° C. Its medical properties are similar to oil of sassafras, after which it is named.

Dose.—0.3 Cc. (5 minims).

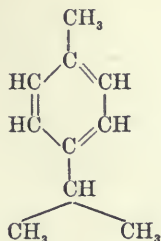
Apiol, $C_6H_3O_2 \begin{matrix} CH_2CH=CH_2 \\ HOCH_3 \\ OCH_3 \\ O > CH_2 \end{matrix}$ is safrol, with two hydrogen atoms of its benzene

nucleus replaced by (OCH₃) groups. It is a constituent of parsley fruit, and is a crystalline solid, melting at 30° C. Commercial apiol, highly esteemed by French physicians as an emmenagogue, is said to be the oleoresin of parsley fruit.

Several hydrocarbons having the formula $C_{10}H_{14}$ are known. The simplest of these are the methyl-propyl-benzenes, $C_6H_4 \begin{matrix} CH_3 \\ \diagup \\ C_3H_7 \end{matrix}$. As all bi-derivatives of benzene are known in three modifications, ortho-, meta-, and para- (see p. 763), so we have ortho-, meta-, and para-methyl-propyl benzene, in which the propyl group is normal, viz.:



More important than these, however, is the para-methyl-isopropyl-benzene, or *cymene*:



This hydrocarbon, thus named because first found in oil of cumin (*Cuminum cyminum*), is made synthetically by treating para-brom-isopropyl-benzene with methyl iodide in the presence of metallic sodium.

The graphic formula of cymene, given above, is proved correct by the synthesis just mentioned, and also by its behavior under various oxidizing agents.

Under certain conditions of oxidation it can be changed to para-toluic acid, $\text{C}_6\text{H}_4\text{CH}_3\text{COOH}$; to terephthalic acid, $\text{C}_6\text{H}_4(\text{COOH})_2$, or to cuminic acid, $\text{C}_6\text{H}_4\text{CH}(\text{CH}_3)_2\text{COOH}$ (1), (4).

It will be noted that in the first-named acid the propyl group is oxidized to the carboxyl group; in the third-named acid it is the methyl group that is converted into the carboxyl, the isopropyl group remaining; while in the second acid both methyl and propyl groups are changed to carboxyl groups. Since all three acids have the groups attached to the benzene nucleus in the para-position, that must be the position of the methyl and propyl groups in the cymene.

Cymene is of little practical importance in pharmacy, but it is of interest as a constituent of several volatile oils, notably, the oils from the various species of thyme.

The following cymene derivatives or analogues are of pharmaceutical importance:

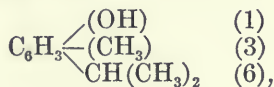
Thymol, U. S. P.

Thymol iodide, U. S. P.

Carvacrol.

Carvone.

Thymol (iso-propyl-metacresol),



is an alcohol closely allied to cymene (compare formula), though the only proof of relationship is the conversion of thymol into cymene by treatment with phosphorous pentasulphide.

THYMOL. Thymol.

$$\text{C}_{10}\text{H}_{14}\text{O} = 148.98$$

A phenol $[\text{C}_6\text{H}_3(\text{CH}_3)(\text{OH})(\text{C}_3\text{H}_7)]$ 1:3:4, occurring in the volatile oil of *Thymus vulgaris* Linné, and in some other volatile oils. It should be kept in well-stoppered bottles.

Large, colorless, translucent rhombic prisms, having an aromatic, thyme-like odor, and a pungent, aromatic taste, with a very slight caustic effect upon the lips.

Its specific gravity as a solid is 1.030 at 25° C. (77° F.), but when liquefied by fusion it is lighter than water. It melts at 50° to 51° C. (122° to 123.8° F.), remaining liquid at considerably lower temperatures. When triturated with about equal quantities of camphor, menthol, or hydrated chloral, it liquefies.

Soluble in about 1100 parts of water at 25° C. (77° F.), and in less than its own weight of alcohol, ether, or chloroform, soluble in glacial acetic acid and fixed and volatile oils.

Its alcoholic solution is optically inactive.

If a very small crystal of Thymol be dissolved in 1 Cc. of glacial acetic acid, and then 6 drops of sulphuric acid and 1 drop of nitric acid be added, the liquid will assume by reflected light a deep bluish-green color.

If 1 Gm. of Thymol be heated in a test-tube, in a water-bath, with 5 Cc. of a 10 per cent. solution of sodium hydroxide, a clear, colorless, or very slightly reddish solution should be formed, which becomes darker on standing, but without the separation of oily drops. If to this solution a few drops of chloroform be added and the mixture agitated, a violet color will be produced.

An alcoholic solution of Thymol should not be colored by ferric chloride T.S.

When a crystal of Thymol is heated in an open dish or in a watch-glass, on a water-bath, it should gradually volatilize, leaving no residue (absence of *inorganic impurities*).

Tests for Identity.—Given above.

Impurities.—Inorganic impurities. Details above.

Remarks.—Thymol occurs in several volatile oils, one of the most abundant sources being oil of horsemint. Thymol is obtained by saponifying oil of horsemint or oil of thyme, or a specific fraction of the same, with sodium hydroxide. The thymol sodium compound dissolves in the aqueous layer, which is separated from the oily portion, and the thymol set free by treating with hydrochloric acid.

It is also made in the manner similar to the manufacture of menthol, that is, by submitting the oil to a freezing mixture and separating the stearopten (thymol) from the eleopten (p. 806) by means of a centrifugal apparatus. Another method of obtaining thymol is by the fractional distillation of the oils mentioned above. Of these three processes, the chemical treatment with sodium hydroxide is the one generally employed. Thymol has also been made synthetically from cuminaldehyde.

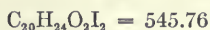
Thymol affords an interesting illustration of crystallization by means of a nucleus. When obtained by the several methods just mentioned it is in the form of the thick liquid, and it is only when a crystal of thymol is introduced that the mass crystallizes. Thymol, when rubbed with some solid substance, such as chloral hydrate, menthol, and camphor, forms a thick liquid. A characteristic test for thymol is the blue-green color produced when a crystal of it is dissolved in glacial acetic acid and treated first with sulphuric acid, then with nitric acid.

Thymol is used as a valuable addition to antiseptic solutions, not only on account of its agreeable odor, but because it possesses marked antiseptic properties.

Dose.—125 milligrammes (2 grains).

Liquor antisepticus, which has already been described on p. 184, owes much of its value to the thymol it contains.

Annidalin was the name originally applied to aristol. Later, however, there was introduced an annidalin differing from aristol in being dithymol tri-iodide.

THYMOLIS IODIDUM. Thymol Iodide.

Dithymol-diiodide $[(\text{C}_6\text{H}_2\text{CH}_3\text{C}_3\text{H}_7\text{OI})_2]$, obtained by the condensation of two molecules of thymol and the introduction of two atoms of iodine into the phenolic groups of the thymol; it contains 45 per cent. of iodine. Thymol Iodide should be kept in amber-colored vials, protected from the light.

A bright, chocolate-colored, or reddish-yellow, bulky powder, with a very slight aromatic odor.

Insoluble in water and glycerin; slightly soluble in alcohol at 25°C . (77°F .); readily soluble in ether, chloroform, collodion, and in fixed and volatile oils, leaving a slight residue.

It is decomposed when heated to the melting-point.

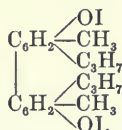
It is not soluble in solution of sodium hydroxide, either cold or warm. When heated with concentrated sulphuric acid, it is decomposed with the separation of iodine.

If 0.1 Gm. of Thymol Iodide be thoroughly ignited in a porcelain crucible, it should leave not more than 0.003 Gm. of residue (limit of ash).

Tests for Identity.—Given above.

Impurities.—Iodides, alkalis, free iodine. Details in Part V. Excess of ash. Details above.

Remarks.—This is the popular *aristol*, or dithymol di-iodide:



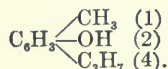
It is made by adding solution of 6 grammes of iodine, 9 grammes of potassium iodide, in 10 grammes of water, to a solution consisting of 5 grammes of thymol in 10 grammes of 13 per cent. sodium hydroxide solution. On mixing, *aristol* separates out in the form of a brown powder.

Aristol is a valuable iodoform substitute, containing the iodine in an easily assimilable form. From the standpoint of the iodine strength, however, it is inferior to both iodoform and iodol, for, as indicated by the formula, $\text{C}_{20}\text{H}_{24}\text{O}_2\text{I}_2$, it contains 46 per cent. of iodine (being $\frac{252}{545.76}$ iodine). The pharmacopœial requirement is that it should contain 45 per cent. of iodine and should yield not more than 3 per cent. of ash.

While the popularity of *aristol* is largely due to the persistent advertising of the firm who hold the monopoly of manufacture, it is a very valuable antiseptic, possessing over the iodoform the great advantage of having an agreeable odor. Some care should be bestowed on the keeping of *aristol*, as it is found that it decomposes when submitted to strong light.

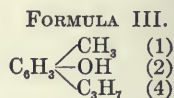
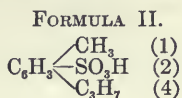
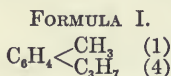
Thymacetin, $\text{CH}_3 > \text{C}_6\text{H}_2 < \text{OC}_2\text{H}_5$, This substance was suggested as a substitution for phenacetin, and is made by treating amido-thymol with ethyl iodide, and treatment of the resulting product, $\text{CH}_3 > \text{C}_6\text{H}_2 < \text{OC}_2\text{H}_5$ with glacial acetic acid. Dose, 5 to 15 grains.

Carvacrol.—An isomeric alcohol of thymol is carvacrol, iso-propyl-ortho-cresol,



A comparison of this formula with that of thymol, given above, shows that the only difference is that in thymol the propyl group is attached to carbon atom number 6 of the benzene nucleus, while in carvacrol it is attached to carbon atom number 4. In both substances the propyl group is of the "iso" variety.

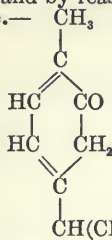
Carvacrol has been made synthetically from cymene by converting the latter (Formula I) into its sulphonic acid derivative (Formula II), and on replacing the acid group by a hydroxyl group, carvacrol (Formula III) is obtained.



This body—a colorless, thick, acid oil—is of little importance in pharmacy, save as a constituent of some volatile oils, notably oil of origanum.

It was first prepared by heating carvone with glacial phosphoric acid, and because so obtained, and by reason of its acidity, it was called carvacrol.

Carvone.—



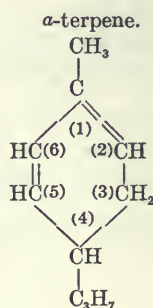
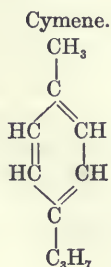
This body is a ketone, and is isomeric with carvacrol. There are two forms of carvone, having the same graphic formula, but differing in that one deviates a ray of polarized light to the right, while the other turns it to the left. Dextrogyre carvone is the flavoring constituent of oil of caraway, *Carum carvi*, whence the name—carvone—is derived; while the levogyre form is a liquid of entirely different odor; that of oil of spearmint, of which it is the important constituent.

TERPENES

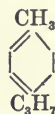
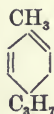
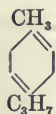
The terpenes are a series of hydrocarbons having the formula $\text{C}_{10}\text{H}_{16}$. While twenty-three of these terpenes are known, a much larger number is possible.

Thus we can imagine a series of chain compounds having the formula, $\text{C}_{10}\text{H}_{16}$, and among those which are known we find *anhydrogeraniol*, the formula of which is supposed to be $(\text{CH}_2)_2\text{C}=\text{CH}-\text{CH}_2-\text{CH}=\text{C}-\text{CH}=\text{CH}_2$.

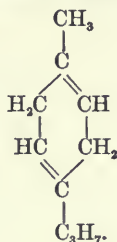
Again, we can have—what is generally the case—a body, $\text{C}_{10}\text{H}_{16}$, representing cymene, with one of its ethylene bonds loosened and with an atom of hydrogen attached to each of the free bonds, viz.:



Since, as mentioned on p. 798, three cymenes containing a normal propyl group are known (ortho-, meta-, and para-), so the closely similar terpenes may be found with their methyl and propyl groups in these three positions. Again, we can imagine a new line of terpenes, depending on whether the propyl group is normal or iso. Again, we can have four separate classes of terpenes dependent upon the relative position of the ethylene bond found in the benzene nucleus, as shown in the following figures:



It is hardly necessary to state that the hexagons just given represent the benzene nucleus, with one double bond loosened. Thus, the first one is:



Beside all these combinations of $C_{10}H_{16}$, we can imagine each one of these compounds, containing asymmetric carbon atoms, existing in stereo-isomeric forms, and thus obtaining levogyre, dextrogyre, and inactive terpenes. Lastly, we can carry the isomeric possibilities still further by imagining terpenes containing, instead of methyl and propyl groups above given, two ethyl groups or one ethyl and two methyl groups. Such forms of terpenes, however, have not yet been isolated.

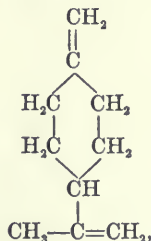
The known terpenes are divided into the following systematic groups:

The Aliphatic Terpenes.—Each molecule of these terpenes absorbs six atoms of bromine, showing the presence of three ethylene groups. Two such terpenes are known, *myricene*, which is found in the oil of bay, and the *anhydrogeraniol*, the formula of which is given on the preceding page, and which is found in the oil of geranium.

The Limonene Group.—In this group of terpenes each molecule absorbs four atoms of bromine, thereby showing the presence of two ethylene bonds.

Among these terpenes we have the three *limonenes* (d, l, and i); *terpinolene*, an artificial terpene obtained by heating oil of turpentine with alcoholic sulphuric acid; *carvestrene* and *thujene* (or *tanacetene*), which are also artificially prepared, and *sylvestrene*, found in Swedish pine-needle oil.

The *terpinene* group consists of two hydrocarbons, *terpinene* and *phellandrene*, the character of which are puzzling. It was formerly thought that neither would absorb bromine, but it is now known that each yields a dibromide. Semmler thinks terpinene is pseudo-limonene, viz.:



while the investigations of Wallach and Herbig indicate that phellandrenes have a structure similar to carvenone.

Among the terpenes belonging to this group we find *terpinene*, found in cardamom oil, dextrogyre *phellandrene*, from the oil of phellandrium, and levogyre phellandrene, found in the oil of *Eucalyptus amygdalina*.

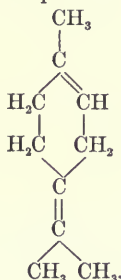
The Pinene Group.—Each molecule of this group takes up two atoms of bromine, showing the presence of one ethylene bond. The known terpenes of this group are *pinene*, found in the oil of turpentine; *camphene*, found in oil of ginger, and made artificially by Berthelot (the only solid terpene known), and *fenchene*, which is an artificial terpene made indirectly from fenchyl alcohol.

Besides the terpenes just classified, the following have been discovered of recent years, and have not as yet been well characterized: fenchylene, euterpene, tricyclene, bornylene, sabinene, an unnamed terpene from resin of *cannabis indica*, and a synthetic terpene from synthetic pulegone, and apparently an ortho-terpene.

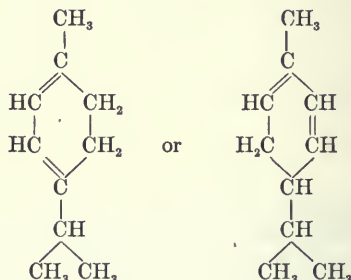
The difference between the different terpenes can best be studied by comparison of a few typical formulas.

Characteristic for the first group given above is the formula of anhydrogeraniol, given on p. 802. Of the second group, the formulas ascribed to terpinolene and limonene are as follows:

Terpinolene.



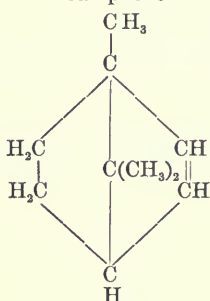
Limonene.



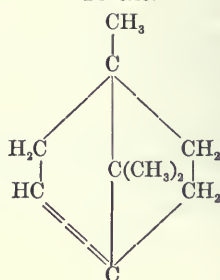
As to limonene, which of the formulas just given is correct is still a matter of controversy.

As mentioned above, the structure of the third group is a matter of doubt, while representatives of the fourth group (camphene and pinene) are supposed to have the formulas given below:

Camphene.



Pinene.



It will be seen in these two formulas the propyl group is found within the benzene nucleus.

The transformation of one terpene into the other is a comparatively simple matter. Thus, on standing, limonene, found in the oil of lemon, is readily converted into pinene. This explains why the oil of lemon becomes terebinthinate.

Terpenes possess many striking chemical characteristics. For instance, those absorbing bromine generally give very beautiful crystalline bromo-compounds.

Another beautiful line of terpene derivatives are the nitroso-chlorides, such as limonene nitroso-chloride, $\text{C}_{10}\text{H}_{16}\text{NOCl}$, and these, on reduction, frequently yield

well-crystallized oximes, such as carvoxime, $C_{10}H_{14}NOH$. Some terpenes are optically active, while others have no effect on the polarized ray of light. Those which do rotate the ray of light to the right or to the left always contain an asymmetric carbon atom (see p. 685).

The subject of terpenes serves as an introduction to that large class—the volatile oils. Practically all volatile oils are mixtures of many chemicals, and the old-fashioned classification of Dumas (1833) into terpenes, oxygenated products, oils containing nitrogen, and those containing sulphur, has long since become antiquated. For this reason the nitrogenated oils and those containing sulphur have already been considered under the appropriate grouping (oils of mustard, see p. 745; oil of bitter almond, see p. 777). Likewise, two other volatile oils that have no connection with the terpenes are the oil of sweet birch and the oil of wintergreen, which are esters of salicylic acid, and have been considered among the derivatives of this chemical. Nor is it wholly correct to consider the rest of the volatile oils merely as terpene derivatives. It is true, in the case of the larger number of them, that most of their bulk consists of terpenes, but it is also true that these oils owe very little of their character to the terpenes. Thus, while oil of lemon consists of about 94 per cent. of the terpene, limonene, this is simply the solvent for the odorous principle, that consisting chiefly of *citral*. In fact, modern chemistry has attempted the problem of obtaining concentrated oils by removing the terpenes, and, in consequence, there is now on the market a large line of the so-called *terpeneless volatile oils*. In many cases these oils are not merely stronger than the natural oil, but are much better for general use. Thus, the terpeneless oil of lemon has, roughly speaking, sixteen times the flavoring power as has the oil of lemon, and possesses the great advantage of not becoming terebinthinate. A very good review of the fallacy of considering the terpenes, or even the terpene derivatives, as the most important constituents of the volatile oils, is found in the admirable paper of Professor Kremers, in the Proceedings of the American Pharmaceutical Association, 1898 (p. 214). This paper shows that the volatile oils contain a large number of different esters, aldehydes, alcohols, and ketones.

Another point in the modern consideration of these derivatives is the contraction of the meaning of the word terpene. It was formerly applied to all the derivatives of cymene from $C_{10}H_{14}$ to $C_{10}H_{20}$. In modern chemical nomenclature bodies of the formula $C_{10}H_{16}$ are called terpadienes, while the word terpene is reserved for bodies of the formula $C_{10}H_{18}$, and the substance of the formula $C_{10}H_{20}$ are called terpanes. The application of the word terpene to $C_{10}H_{18}$ is unfortunate, since the word has for years been associated with bodies having the formula $C_{10}H_{16}$. Of the two classes last mentioned, terpanes represent the body $C_{10}H_{18}$, with one of their double bonds loosened and the bonds thus freed taken up with hydrogen atoms, making $C_{10}H_{18}$, while in the terpanes both double bonds are similarly loosened, and taken up by hydrogen, making $C_{10}H_{20}$.

Interesting modifications of the formula $C_{10}H_{16}$ are found in a group of hydrocarbons having the formula C_5H_8 . These bodies are called *hemiterpenes*; literally, "half-terpene." An example of such a hemiterpene is *isoprene*, obtained by the distillation of rubber. Again, we have bodies that are modifications of terpenes, containing half as much again carbon and hydrogen as terpenes, and, therefore, having the formula $C_{15}H_{24}$. This class of hydrocarbons are called *sesquiterpenes*. As examples of this may be cited *caryophyllene*, found in oil of cloves, *cadinene*, found in oil of cade, and *zingiberene*, found in oil of ginger. Bodies having formula $C_{20}H_{32}$, $C_{26}H_{48}$, and $C_{40}H_{64}$ are also known.

Two words of the obsolete nomenclature of the volatile oils still used in pharmacy are the terms *eleoptene* and *stearoptene*. These words were coined by Berzelius to represent respectively the liquid and solid portions of volatile oils. As mentioned above, most of the volatile oils are mixtures of quite a number of substances, and it so happens in some cases that some of these substances are solids dissolved in the liquid portion. These solid substances found in oils can sometimes be removed by chilling the oil in a freezing mixture and draining off the unfrozen eleoptene. Among the stearoptenes are found three official substances, camphor, menthol, and thymol. In modern chemistry the question as to whether the constituent of a volatile oil is solid or liquid is of but little importance, and the word stearoptene is not used in the new pharmacopœia.

Beside the official volatile oils containing terpenes, there are several terpene derivatives of interest to pharmacy which can be considered in a purely chemical sense. These are as follows:

Hydrocarbon:	Terebene, U. S. P.
Monatomic alcohols:	Terpineol. Menthol, U. S. P. Borneol.
Diatomic alcohol:	Terpin hydrate, U. S. P.
Aldehyde:	Citral.
Ketone:	Camphor, U. S. P.
Dibasic acid:	Camphoric acid, U. S. P.
Inner oxide:	Eucalyptol, U. S. P.

TEREBENUM. Terebene.

A liquid consisting of dipentene and other hydrocarbons, obtained by the action of concentrated sulphuric acid on oil of turpentine and subsequent rectification with steam.

Terebene should be kept in well-stoppered bottles, in a cool place, protected from light.

A colorless, thin liquid, having a rather agreeable, thyme-like odor, and an aromatic, somewhat terebinthinate taste.

Specific gravity: about 0.850 at 25° C. (77° F.).

Only slightly soluble in water, but soluble in three times its volume of alcohol.

It boils at 155° to 165° C. (311° to 329° F.).

On exposure to light and air, Terebene gradually becomes resinified, and acquires an acid reaction.

Terebene should possess its characteristic agreeable odor, should not redden moistened blue litmus-paper (absence of *acids*), and should be completely inactive toward polarized light (absence of *unaltered oil of turpentine*).

If about 10 Cc. of Terebene be evaporated in a porcelain dish, on a water-bath, not more than a very slight residue should be left (absence of more than traces of *resinous substances*).

Tests for Identity.—Given above.

Impurities.—Acids, unaltered oil of turpentine, excess of resinous matter. Details above.

Remarks.—Terebene is a mixture of three terpenes: dipentene, pinene, and terpinene. Terebene is manufactured by treating 20 parts of oil of turpentine with 100 parts of concentrated sulphuric acid, and allowing the mixture to stand twenty-four hours. It is then heated to boiling, allowed to cool, when the oily layer on top is separated and neutralized with calcium carbonate and then dis-

tilled. The distilled terebene is rectified in order to secure a pure product.

Terebene in alcohol solution (1 : 500) is used as an antiseptic. It is also used in the concentrated form for bronchial troubles, for which purpose it is best given in the form of capsules. When administered for too long a time, however, it is apt to cause irritation of the stomach.

Dose.—0.5 Cc. (8 minims).

Terpineol, $C_{10}H_{17}OH$.—This unofficial chemical is made by the treatment of oil of turpentine with sulphuric acid and nitric acid. Several terpineols are known, three of them being solids and two viscid liquids. The crystalline terpineol is found in commerce, melts at $35^{\circ}C$., and was first obtained from the liquid terpineol by a method which afforded a striking illustration of the possibilities of crystallization. The liquid had been known to chemists, and many efforts have been made to secure its crystallization. All these attempts, such as freezing and the use of the nuclei, etc., have been in vain until Bouchardat and Voiry applied the idea of crystallization by analogy. These chemists had already prepared from India-rubber, by indirect means, a crystalline body having the formula $C_{10}H_{17}OH$, which they called caoutchcin hydrate, and a minute crystal of this they dropped into liquid terpineol well cooled by use of a freezing mixture, and immediately the liquid terpineol began to crystallize, and in a short time five-sixths of the mass was solid. From that time the manufacture of crystalline terpineol has been simple, all that was necessary being to add to subsequent batches a few crystals of terpineol, such as originally produced by this brilliant experiment.

Terpineol is not used in medicine, but is largely used in perfumery as the basis of lilac perfumes; the substance in diluted form possesses an agreeable odor resembling that of the lilac blossoms. For perfumery purposes the viscid liquid terpineol, frequently marketed as "lilacin," is preferable to the crystalline.

TERPINI HYDRAS. Terpin Hydrate.



The hydrate $[C_{10}H_{18}(OH)_2 + H_2O]$ of the diatomic alcohol terpin. Terpin Hydrate should be kept in well-stoppered bottles.

Colorless, lustrous, rhombic prisms, nearly odorless, and having a slightly aromatic and somewhat bitter taste. Permanent in the air.

Soluble in about 200 parts of water and in 10 parts of alcohol at $25^{\circ}C$. ($77^{\circ}F$.); in 32 parts of boiling water and in 2 parts of boiling alcohol; also soluble in about 100 parts of ether, 200 parts of chloroform, or 1 part of boiling glacial acetic acid.

Terpin Hydrate melts when quickly heated at 116° to $117^{\circ}C$. (240.8° to $242.6^{\circ}F$.), with the loss of water, and, at the temperature of boiling water, sublimes in fine needles.

When heated in a flask adapted for distillation, it first loses water. At $258^{\circ}C$. ($496.4^{\circ}F$.) anhydrous terpin distils over without decomposition, soon solidifying to a crystalline, hygroscopic mass, which melts at 102° to $105^{\circ}C$. (215.6° to $221^{\circ}F$.). When strongly heated on platinum foil, it burns with a bright, smoky flame, leaving no residue.

If to its hot, aqueous solution a few drops of sulphuric acid be added, the liquid will become turbid and develop a strongly aromatic odor.

Terpin Hydrate should not have the odor of turpentine, and its hot, aqueous solution should not redden blue litmus-paper.

Tests for Identity.—Just given.

Remarks.—Terpin hydrate is the crystalline hydrate of terpin, $C_{10}H_{18}(OH)_2$, which in turn can be considered as terpineol, with which one molecule of water has been combined or as a terpene from oil of turpentine (pinene, for example), to which two molecules of water have been added.

The water is, of course, added by chemical means—treatment with diluted mineral acids:

Oil turpentine (or its pinene).....	$C_{10}H_{16}$
Add water.....	HOH
And we get terpineol.....	$C_{10}H_{17}OH$
Add another water.....	$H\ OH$
And we get terpin. <i>hydrate</i>	$C_{10}H_{18}(OH)_2$

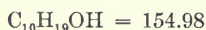
Terpin hydrate is generally manufactured by treating an alcoholic solution of oil of turpentine with nitric acid. The mixture is placed in a shallow porcelain dish for three or four days, when the crystals of terpin hydrate appear. The liquid is drained from the same, and the crystals are dried by pressing between sheets of filter-paper. In order to obtain the pure product the substance is recrystallized. The yield of terpin hydrate by this process is about 12 per cent. of the turpentine used.

Terpin hydrate is used in chronic bronchitis and other troubles of the throat.

Dose.—125 milligrammes (2 grains).

Of late years an elixir of terpin hydrate has become quite popular as a cough remedy. Since the chemical does not dissolve in water, the preparation of an elixir which will contain three grains of terpin hydrate to the teaspoonful is a matter of some difficulty. In fact, about the only way to hold the terpin hydrate in solution is by making the preparation strongly alcoholic. A satisfactory recipe for the elixir is found in the National Formulary. It, however, contains only 1 grain of terpin hydrate to the teaspoonful.

MENTHOL. *Menthol.*



A secondary alcohol $[C_6H_9(CH_3)(OH)(C_3H_7) 1:3:4]$, obtained from the oil from *Mentha piperita* Linné, or other peppermint oils. Menthol should be kept in well-stoppered bottles, in a cool place.

Colorless, acicular or prismatic crystals having a strong and pure odor of peppermint, and a warm, aromatic taste, followed by a sensation of cold when air is drawn into the mouth.

Menthol is only slightly soluble in water, but imparts to the latter its odor and taste. It is freely soluble in alcohol, ether, and chloroform.

It melts at $43^\circ C.$ ($109.4^\circ F.$) to a colorless liquid, boils at $212^\circ C.$ ($413.6^\circ F.$), and volatilizes slowly at the ordinary temperature.

When it is triturated with about an equal weight of camphor, thymol, or hydrated chloral, the mixture becomes liquid.

Its alcoholic solution is neutral to litmus-paper and is levogyrate.

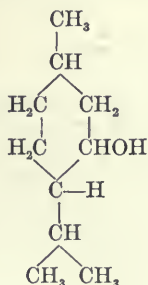
If a little menthol be heated in an open dish, on a water-bath, it should gradually volatilize without leaving any residue (absence of *war*, *paraffin*, or *inorganic substances*).

If a few crystals of Menthol be dissolved in 1 Cc. of glacial acetic acid, and then 3 drops of sulphuric acid and 1 drop of nitric acid added, no green color should be produced (absence of *thymol*).

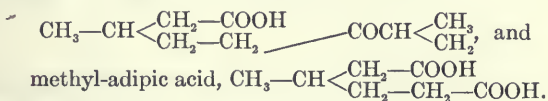
Tests for Identity.—Given above.

Impurities.—Wax, paraffin, inorganic matter, thymol. Details above.

Remarks.—Menthol represents the alcohol of the hydrocarbon menthene, $C_{10}H_{18}$, its graphic formula being as follows:



This formula is proved by its behavior when oxidized with potassium permanganate, when it yields oxymenthylic acid.



That menthol is a secondary alcohol is shown by its oxidation by chromic acid into the ketone, menthone, $C_{10}H_{18}O$.

As mentioned in the official definition, menthol is a secondary alcohol ("a stearopten having nature of a secondary alcohol," U. S. P. 1890), found in the oil of peppermint, and is obtained by placing the oil in a freezing mixture, when the menthol should crystallize out.

In the manufacture of menthol from the oil of peppermint the semisolid mass obtained by freezing is thrown on a filter-paper, and after the greater portion of the liquid is drained, the crystals are dried by placing between folds of filter-paper.

This process affords the test for the purity of oil of peppermint, and the pharmacopœia of 1890 gave it among the tests. Unscrupulous distillers remove the menthol from the oil of peppermint and market the liquid part as oil of peppermint. Such an oil yields no menthol when chilled.

Besides the official tests just given, a pretty reaction identifying the menthol is the treatment of menthol with 50 per cent. sulphuric acid, and boiling the mixture, when a blue coloration results.

Menthol, as thymol, becomes liquid when triturated with an equal weight of camphor or chloral hydrate.

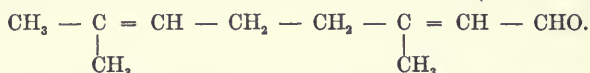
Menthol is used as a refrigerant. Its chief use, however, is as the constituent of antiseptic combinations, particularly those used for spraying in the nose. Another use for menthol is in the manufacture for the menthol pencils used for headache. Menthol rubbed on any part of the body evaporates, producing a chilling of the part, and thus involving a mild form of local anesthesia. It is also used in the form of an inhaler, this consisting of glass tubes plugged with cotton at either end, with intervening space loosely filled with menthol crystals, thus affording free access for the passage of air. When the

air is sucked through this tube, a certain portion of the menthol volatilizes and enters the throat and nose, where it eventually condenses, thus bringing the healing antiseptic menthol in direct contact with the inflamed surface.

Dose.—65 milligrammes (1 grain).

Citral (Geranial), $C_{10}H_{16}O$.—This aldehyde was isolated from oil of lemon in 1888, and found to be the flavoring principle of the oil. Semmer proved it to be identical with geranial, the aldehyde prepared by him by oxidation of geraniol. It is prepared from oil of lemon, which contains from 6 to 8 per cent. of the chemical, and particularly from oil of lemon grass (*Andropogon citratus*), which contains from 70 to 80 per cent., by formation of crystallization compound by treatment of the oil with sodium bisulphite. This compound, after washing with alcohol and ether, is treated with sodium carbonate, and the citral thus freed purified by distillation in vacuo.

As mentioned above, this body is the basis of terpeneless oil of lemon. Chemically, citral is supposed to be the aldehyde of the olefinic hydrocarbon anhydrogeraniol, with formula—

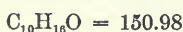


On condensation of this body with acetone there is produced a ketone, pseudo-ionone ($C_{13}H_{20}O$), $\text{CH}_3 - \text{C} = \text{CH} - \text{CH}_2 - \text{CH}_2 - \text{C} = \text{CH} - \text{CH} = \text{CH} - \text{CO} - \text{CH}_3$.

$\begin{array}{c} \text{CH}_3 \\ | \\ \text{CH}_3 - \text{C} = \text{CH} - \text{CH}_2 - \text{CH}_2 - \text{C} = \text{CH} - \text{CH} = \text{CH} - \text{CO} - \text{CH}_3 \\ | \\ \text{CH}_3 \end{array}$
This, on boiling with dilute sulphuric acid and trace of glycerin, yields an isomeric ring compound which is called *Ionone*, and is of great commercial value as the basis of synthetic violet perfume. This is now known to consist of two isomeres, alpha-ionone and beta-ionone.

The real flavor of violet and of orris is still another isomere, called *irone*.

CAMPHORA. Camphor.



The dextrogyrate modification of the saturated ketone [$C_9H_{14}CO$], obtained from *Cinnamomum Camphora* (Linné) Nees et Ebermeier (Fam. *Lauraceæ*), and purified by sublimation. Camphor should be kept in well-closed vessels, in a cool place.

White, translucent masses, of a tough consistence and a crystalline structure, readily pulverizable in the presence of a little alcohol, ether, or chloroform; having a penetrating, characteristic odor, and a pungent, aromatic taste.

Specific gravity: 0.990 at 25° C. (77° F.).

It is optically active, being dextrogyrate.

Very sparingly soluble in water, but readily soluble in alcohol, ether, chloroform, carbon disulphide, petroleum benzin, and in fixed and volatile oils.

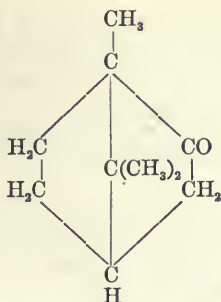
When camphor is triturated, in about molecular proportions, with menthol, thymol, phenol, or hydrated chloral, liquefaction ensues. It melts at 175° C. (347° F.), boils at 204° C. (399.2° F.), and is inflammable, burning with a luminous, smoky flame.

On exposure to the air, it evaporates more or less rapidly at ordinary temperatures, and, when moderately heated, it sublimes, without leaving a residue.

Tests for Identity.—Given above.

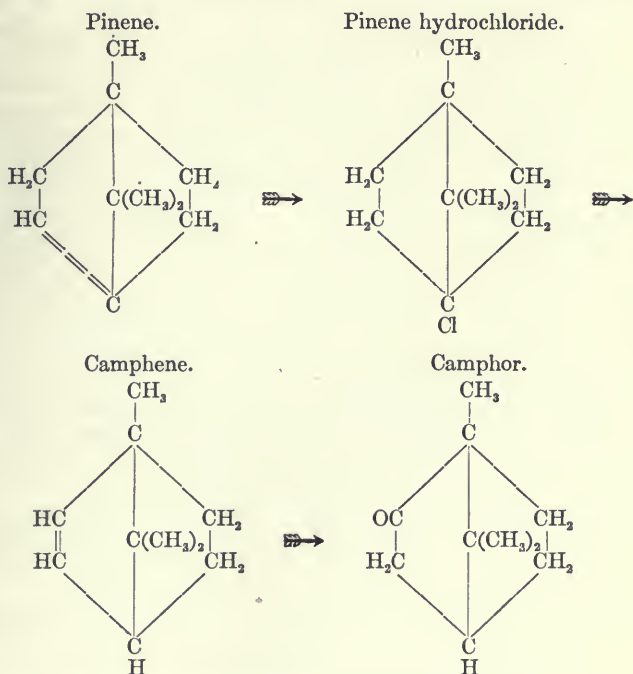
Impurities.—Chlorinated products. Test same as found under benzaldehyde, only a lump of camphor is burned direct.

Remarks.—The constitution of camphor is supposed to be expressed by the following formula:



While menthol is alcohol, camphor, as noted above, is ketone, the oxygen not being combined with hydrogen, but directly attached to a carbon atom.

Camphor has been made synthetically from pinene, the successive stages of the process being given below by means of graphic formulas without any attempt at descriptive detail.



This synthesis of camphor is of interest entirely from a theoretic point of view, it having been a valuable aid in establishing the structural formula of the substance. Several other syntheses have been patented, but none are as yet of technical importance, all the camphor used being from the natural source.

As mentioned above, camphor is obtained from *Cinnamomum camphora*, a handsome tree growing in Formosa. This tree is cut down, and the roots, trunk, and branches cut into small chips, placed in a rude retort containing heated water. When this water is boiled,

the volatile products of the camphor wood distil over. The semisolid distillate is then submitted to hydraulic pressure, which removes all the eleoptene, the stearoptene remaining in the press. The manufacture of this substance affords an interesting and about the only illustration of a volatile oil containing so large a preponderance of the stearoptene as to be semisolid.

The liquid portion separated from the camphor by pressure is comparatively small in quantity, and is marketed under the name of *oil of camphor*, being in considerable demand in the Orient, although its consumption in this country is insignificant. It is well to bear in mind that this oil of camphor is not the familiar camphorated oil (liniment of camphor).

The crude camphor that is separated by expression is mixed with quicklime (to absorb water) and sublimed from a cast-iron retort into a cooled chamber, similar to that used in making flower of sulphur. The camphor in this case condenses into a fine powder, which is then compressed into cakes.

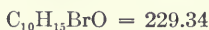
As already mentioned on p. 117, the powdering of camphor is accomplished by the addition of a small quantity of alcohol, an example of pulverization by intervention. The powder thus obtained has the disadvantage of eventually forming again into a hard cake. Camphor, however, can be kept in the form of a permanent powder by triturating the powder, when freshly made, with a minute trace of white vaseline. Such powdered camphor, of course, would be unfit for preparing solutions, but for the usual purpose for which powdered camphor is employed, the addition of a minute quantity of the harmless substance, white vaseline, does not vitiate the camphor. When a lump of camphor, or powdered camphor free from oil, is placed in water, it floats on the surface, and then very rapidly moves to and fro on the surface of the water. This movement is produced by rapid evaporation of the camphor in contact with the water, and the sudden chilling of the water where the evaporation takes place, causing the movement in the direction where the water is warmer.

In the official description of camphor given above it will be noted that the camphor liquefies not only with menthol, but also with thymol and chloral hydrate; the liquefaction taking place in this case is not supposed to be due to chemical change, but a mutual solution of the two solids, with the formation of a liquid. And that no chemical change is produced is shown by the fact that such a liquid, when poured into water, is rapidly separated into the two constituents.

Camphor is a mild stimulant and is somewhat diaphoretic. It is used chiefly as anodyne, both internally and externally.

Dose.—125 milligrammes (2 grains).

CAMPHORA MONOBROMATA. Monobromated Camphor.



A substitution product of camphor [$\text{C}_{10}\text{H}_{15}\text{Br.CO}$].

Colorless, prismatic needles or scales, having a mild but characteristic camphoraceous odor and taste, permanent in the air, unaffected by light, and neutral to litmus-paper.

Almost insoluble in water; freely soluble in alcohol, ether, chloroform, hot petroleum benzin, and fixed and volatile oils; slightly soluble in glycerin; it is also

soluble, without decomposition, in cold, concentrated sulphuric acid, from which it separates again unaltered when the solution is poured into water.

It melts at 76°C . (168.8°F .) and sublimes at a slightly higher temperature. At 274°C . (525.2°F .) it boils without decomposition, and is finally volatilized without leaving a residue.

If a few crystals of Monobromated Camphor be fused in a dry test-tube with metallic sodium, the residue dissolved in water, and the solution acidulated with nitric acid, a copious, faintly yellowish precipitate should be produced upon the addition of silver nitrate T.S.

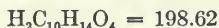
Tests for Identity.—Given above.

Remarks.—This camphor derivative was discovered by Swarts in 1861, who prepared it by heating camphor and bromine for three hours in a sealed tube on a water-bath. It is generally made by the Maisch process, by mixing bromine and camphor in a retort, heating the mixture to 132°C ., then cooling, and extracting the monobromated camphor with benzene. The substance thus obtained is purified by recrystallization:

Monobromated camphor is used as a nervous sedative.

Dose.—125 milligrammes (2 grains).

ACIDUM CAMPHORICUM. Camphoric Acid.



A dibasic organic acid $[\text{C}_{10}\text{H}_{14}(\text{COOH})_2]$, obtained by the oxidation of camphor. Colorless, odorless, monoclinic prismatic crystals or plates; melting at 187°C . (368.6°F .); at a higher temperature yielding an anhydride, and ultimately decomposing without leaving any weighable residue.

Soluble in 125 parts of water at 25°C . (77°F .) and in 10 parts of boiling water; readily soluble in alcohol, less soluble in ether and chloroform; soluble in fatty oils.

Camphoric Acid is dextro-rotary, showing in 10 per cent. alcoholic solution the value $[\alpha]_{\text{D}} = +47.8^{\circ}$.

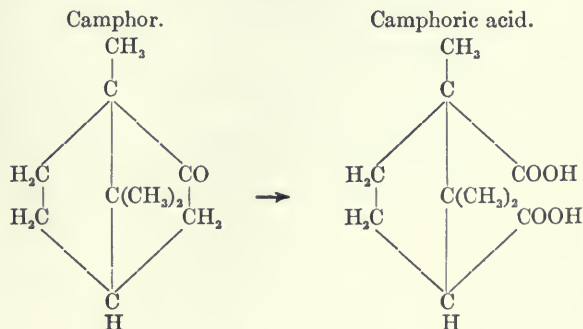
The aqueous solution reddens blue litmus-paper.

Tests for Identity.—Given above.

Impurities.—Nitric acid. Details in Part V.

Remarks.—As mentioned in the pharmacopœial definition given above, camphoric acid is an oxidation product of camphor, nitric acid being the oxidizing agent employed. This accounts for the possible presence of nitric acid as an impurity, as suggested by the pharmacopœia.

The oxidation from camphor to camphoric acid can be best explained by the following graphic formulas:



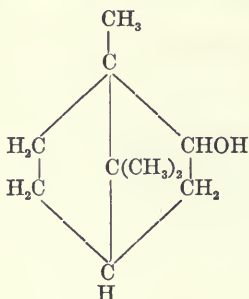
In passing, it may be well to state that the abbreviation, $[\alpha]_D = +47.8^\circ$, given above in the official description, means that the specific rotating power of camphoric acid is 47.8° —that is, when examined in a polariscope its solution, under ideal conditions, rotates the ray of light 47.8° to the right.

To convert ordinary conditions of observation to the ideal, it is necessary to use as data in the calculation the observed angle of rotation, the length of the polariscope tube holding the liquid, and the strength of the solution employed. (For details, see U. S. P. VIII., pp. 580, 581.)

Medical Properties.—Camphoric acid is used like camphor, but it is less irritating.

Dose.—1 Gm. (15 grains).

Borneol, $C_{10}H_{17}OH$.—This is a variety of camphor obtained from the plant, *Dryobalanops Camphora*, and is used quite largely in the east, although not employed in Europe and America. It can be made by treating camphor with sodium alcoholate, and investigations prove that it possesses the following graphic formula:



It differs from camphor in having two extra atoms of hydrogen and in being an alcohol, not a ketone.

EUCALYPTOL. Eucalyptol.

$$C_{10}H_{18}O = 152.98$$

An organic oxide (cineol), obtained from the volatile oil of *Eucalyptus Globulus* Labillardière (Fam. *Myrtaceæ*) and from other sources. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless liquid, having a characteristic, aromatic, and distinctly camphoraceous odor, and a pungent, spicy, and cooling taste.

Specific gravity: 0.925 at 25°C . (77°F .).

Soluble in all proportions in alcohol.

Boiling-point: 176° to 177°C . (348.8° to 350.6°F .).

Eucalyptol is optically inactive (distinction from the oil of *eucalyptus* and many other volatile oils).

When exposed to a temperature somewhat below 0°C . (32°F .) it solidifies to a mass of colorless, needle-shaped crystals, which liquefy at -1°C . (30.2°F .).

If 1 Cc. of Eucalyptol be placed in a freezing mixture, and an equal volume of phosphoric acid be gradually added, a solid white crystalline mass of cineol-phosphoric acid should result, and if warm water be then added, the cineol will separate.

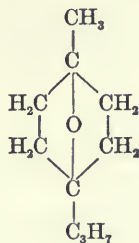
If 5 Cc. of Eucalyptol be shaken with 5 Cc. of sodium hydroxide T.S., it should not diminish in volume.

Its alcoholic solution should be neutral to litmus-paper, and if to 5 Cc. of this solution a drop of ferric chloride T.S. be added, there should not be produced a brownish or violet color (absence of *phenols*).

Tests for Identity.—Given above.

Impurities.—Oil of eucalyptus and other oils, phenols. Details above.

Remarks.—Eucalyptol was originally called *cineol*, because discovered in the oil of the seed of *Artemisia cinæ*, a variety of Levant wormseed. Eucalyptol is made synthetically by treatment of terpin with diluted sulphuric acid, and this synthesis suggests that eucalyptol is the anhydride of terpin, with the following graphic formula:



On closely examining this formula it is found of interest, inasmuch as the oxygen is placed on the interior of the benzene nucleus. The formula is thus expressed, because while the compound contains oxygen, the substance gives none of the reactions usually obtained from organic chemicals containing oxygen, such as alcohols, aldehydes, ketones, or esters. Eucalyptol is, therefore, supposed to represent one of a unique chemical group called inner oxides.

Eucalyptol is prepared commercially by the fractional distillation of oil of eucalyptus; that portion of the oil distilling between 130° and 180° C. being collected and purified by redistillation over potassa. It is used as an antiseptic and stimulant, and is a constituent of the official Antiseptic Solution.

Dose.—0.3 Cc. (5 minims).

Artificial Musk.—Mention might here be made of the several chemicals, the odor of which so closely resembles that of musk that they can be used in perfumery in place of the natural product.

Of the many varieties, the most important is *musk Baur*, which consists either of trinitrobutyltoluol, $\text{C}_6\text{H} \begin{smallmatrix} \text{CH}_3 \\ \diagup \text{C}_4\text{H}_5 \diagdown \\ (\text{NO}_2)_3 \end{smallmatrix}$, or trinitrobutylxylol, $\text{C}_6 \begin{smallmatrix} (\text{CH}_3)_2 \\ \diagup \text{C}_4\text{H}_3 \diagdown \\ (\text{NO}_2)_3 \end{smallmatrix}$.

These are made by preparing the appropriate butyl compound by treating toluene or xylene with butyl bromide in the presence of aluminum chloride, and then treating the butyl compound with nitric acid.

Musk Baur occurs in white crystals, soluble in 200 parts alcohol, and this saturated alcoholic solution has perfume strength about equivalent to the official (5 per cent.) tincture of musk.

Too much stress cannot be laid on the fact that the similarity in odor is purely accidental, and that under no circumstances can the artificial be used for the natural musk in medicine.

CHAPTER XLVII

VOLATILE OILS AND VOLATILE OIL DRUGS

A LARGE number of volatile oils and drugs depending for their activity upon volatile oils are recognized by the pharmacopœia, and these we will study in the following order:

- I. Official volatile oils obtained from unofficial drugs.
- II. Official drugs containing volatile oil.
- III. Official drugs containing volatile oil and resin.
- IV. Official drugs containing volatile oil and bitter principle.
- V. Official oleoresins.
- VI. Official balsams.

Official volatile oils not considered in the first group will be studied under the drug whence they are derived.

OFFICIAL VOLATILE OILS OBTAINED FROM UNOFFICIAL DRUGS

The chemistry of volatile oils has been studied on p. 805, but before taking up the individual members of the group a few words as to the physical difference between fixed and volatile oils may be advisable. The general test for distinction—in fact, expressed in the names “fixed” and “volatile”—was touched upon in discussing fixed oils (p. 723).

Drop some of the liquid on a piece of paper, and warm the stain over a lamp; if a volatile oil, the stain disappears; if a fixed oil, it remains.

A very homely test is the behavior of a glass stopper toward fixed and volatile oils. The ground-glass stopper of a bottle containing a fixed oil rotates easily and quietly in the neck, whereas a volatile oil stopper, when twisted in the neck, makes a squeaking noise under the operation.

The **separation of volatile oils** from their original sources varies according to the nature of oil, the character of the plant part whence derived, and other considerations. The methods can, however, be roughly classified into the following groups:

I. *Distillation with Water*.—In this case the drug is placed in a still with cold water, care being taken to prevent the drug from touching the bottom of the still, for if so exposed, the plant part is apt to scorch, thus vitiating the odor of the distillate (p. 102). The water is then slowly and carefully heated, and as it distils it carries over with it the volatile oil found in the plant. This is the process by which most crude oils are distilled in their native home, especially those distilled in the Orient. Example, oil of cajuput.

II. *Distillation with Steam*.—This process has already been fully described (p. 103), hence it is merely necessary to say that it is employed in place of distillation with water by all progressive distillers of oils produced in Europe and America. This is the way that oil of peppermint is now obtained in Michigan.

III. *Distillation Alone*.—This process is applied in the case of some crude cheap oils, such as the manufacture of oil of turpentine from its oleoresin, which is placed in a still and cautiously heated

until all the oil passes over. The residue in the still in the case considered constitutes official rosin. Even in the case of oil of turpentine, some distillers introduce a small stream of water into the distilling mass to lessen the danger of scorching.

IV. *Destructive Distillation*.—The so-called oils obtained by this process, which has already been described on p. 103, are in truth tarry products, and no longer classed among the true oils. An example of these is the official oil of cade.

V. *Vacuum Distillation*.—This process, described on p. 104, is practically never used in obtaining a crude oil from its plant sources, but is used largely in refining crude oils.

VI. *Expression*.—The oils from the rind of the fruits of the Citrus family—lemon, orange, bergamot, etc.—reside in the outer layer of cells, hence can be obtained by expression. This process is used in these cases because the distillation of the rind yields an inferior oil. The extraction of the oil is accomplished either by pressing the quarter or half fruit against a sponge, which is eventually expressed into the container, or by the use of a complicated machine which ruptures the oil-cells by scraping, and then pressing the broken cells against sponges. This machine works well only with the round fruits, orange and bergamot, being useless in the case of lemon.

The *ecuelle à piquer* is a hollow bowl disk studded with needles on its concave side, and provided with a hollow handle which collects the oil separated from the rind by the pricking of needles when the instrument is revolved against the fruit. It is not used much at present.

VII. *Enfleurage*.—This method is employed in removing from flowers odors too subtle to stand distillation and too scarce to permit of expression. Briefly explained, the process consists of bringing the fresh flowers in contact with lard made odorless by prolonged washing with water. The lard is spread on glass plates in thin layers, and thereon are thrown the flowers, usually prevented from direct contact with the lard by having wire netting interposed. When the flowers wither, new ones are spread on the netting, and the batches are renewed until the lard has the sufficient amount of odor. Such odorous lard is called a *pomade*, the qualifying phrases *triple* and *quadruple* meaning that the lard has been impregnated with the odor of three or four times its weight of flowers respectively. The pomades are usually made direct into perfume by mixing the lard with cologne spirit (p. 655), which dissolves the perfume, but not the fat. The latter is then separated by filtration. The filtrate forms the perfumer's *extract*, the standard strength of which is the filtrate from a mixture of 1 kilo of pomade and 1250 Cc. cologne spirit. While the odorous principles of flowers are known to be volatile oils, only recently have efforts been made to isolate the oil, perfumers finding that the extracts above described answered their purpose. The volatile oil of jasmin has, however, been isolated by Verley by shaking the pomade with liquid petrolatum, which absorbs the oil, and extracting the latter from its petroleum solution by shaking with acetone.

VIII. *Percolation*.—Of late, perfume chemists have been extracting floral volatile oils more or less associated with solid fats by percolating the flowers with low-boiling solvents, such as methyl chloride. It is

beyond the limits of this work to give more than this brief statement as to extraction of volatile oils, and for further details the reader is referred to the books on volatile oils and perfumes cited in the introduction (see Preface).

OLEUM CAJUPUTI. Oil of Cajuput.

A volatile oil distilled from the fresh leaves and twigs of *Melaleuca Leucadendron* Linné (Fam. *Myrtaceæ*), yielding, when assayed by the process given on p. 826, not less than 55 per cent., by volume, of cineol. It should be kept in well-stoppered, amber-colored bottles, in a cool place.

A thin, colorless or greenish liquid, having a peculiar, agreeable, distinctly camphoraceous odor, and an aromatic, slightly bitter taste.

Specific gravity: 0.915 to 0.925 at 25° C. (77° F.).

It is miscible with alcohol in all proportions, also soluble in 1 part of 80 per cent. alcohol. The alcoholic solution should be neutral.

Oil of Cajuput is lævogyrate; the angle of rotation should not exceed -2° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Tests for Identity.—Given above.

Impurities.—Copper. Details in Part V.

Assay for Cineol.—Details given under Oil of Eucalyptus (p. 820).

Composition.—Cineol (p. 815), 55 per cent., a solid terpineol, small amount of terpenes, aldehydes, etc.

Remarks.—The green color of most cajuput oil is due to the copper absorbed from the still.

Medical Properties.—Stimulant and rubefacient.

Dose.—5 Cc. (8 minims).

OLEUM CHENOPODII. Oil of Chenopodium.

A volatile oil distilled from *Chenopodium anthelminticum* Linné (Fam. *Chenopodiaceæ*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A thin, colorless or yellow liquid, having a peculiar, penetrating, somewhat camphoraceous odor, and a pungent and slightly bitter taste.

Specific gravity: about 0.965 to 0.985 at 25° C. (77° F.).

It should be soluble in 5 volumes of 70 per cent. alcohol.

It is lævogyrate; the angle of rotation should not exceed -5° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Tests for Identity.—Given above.

Composition.—A terpene, $C_{10}H_{16}$, liquid substance, $C_{10}H_{16}O$.

Remarks.—The "angle of rotation," mentioned in the official definition given above, refers to the deflection of the ray of light when passing through 100 millimeters of the oil contained in the tube of a polarizing apparatus (p. 684). Explanatory of the measurement of the angle in this case (-5°), it may be stated that the minus sign means a rotation to the left (lævogyrate), while the 5° of rotation is based on the standard 360° of the circle. If the substance rotated the ray of light 5° to the right, it would be expressed " $+5^{\circ}$."

This statement as to the angle of rotation is here given not only to explain this particular case, but also because similar data are given regarding many of the oils of the present pharmacopœia.

Medical Properties.—Anthelmintic, usually administered mixed with castor oil.

Dose.—0.2 Cc. (3 minims).

Chenopodium, or *American wormseed*, was official in the pharmacopœia of 1890, but was dropped in the last revision. Its only important constituent is the oil described above.

OLEUM ERIGERONTIS. Oil of Erigeron.

A volatile oil distilled from the fresh flowering herb of *Erigeron canadensis* Linné (Fam. *Compositæ*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A pale yellow, limpid liquid, rapidly becoming darker and thicker by age and exposure to the air, having a peculiar, aromatic, persistent odor, and an aromatic, slightly pungent taste.

Specific gravity: 0.845 to 0.865 at 25° C. (77° F.).

Soluble in an equal volume of alcohol (distinction from *oil of fireweed* derived from *Erechthites hieracifolia* Rafinesque (Fam. *Compositæ*) and from *oil of turpentine*).

It is dextrogyrate, the angle of rotation being about +50° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Tests for Identity.—Given above.

Impurities.—Oil of fireweed and oil of turpentine. Details above.

Composition.—Dextrogyrate, limonene, etc.

Medical Properties.—Hemostatic and stimulant.

Dose.—1 Cc. (15 minims).

OLEUM JUNIPERI. Oil of Juniper.

A volatile oil distilled from the fruit of *Juniperus communis* Linné (Fam. *Coniferae*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless, faintly green or yellow liquid, having the characteristic odor of juniper, and a warm, aromatic, somewhat terebinthinate and slightly bitter taste.

Specific gravity: 0.860 to 0.880 at 25° C. (77° F.).

Soluble in 10 volumes of 90 per cent. alcohol.

Tests for Identity.—Given above.

Composition.—Pinene, cadinene, etc.

Medical Properties.—Stimulant, diuretic. *Gin* (p. 180) is an alcoholic solution of this oil prepared by distilling fermented juniper berries, and is simulated in the official *compound spirit of juniper* (p. 179). The simple *spirit of juniper* (p. 179) is also official.

Dose.—Of oil, 0.2 Cc. (3 minims).

Juniper berries, the fruit from which the above oil is derived, was official until the revision of 1890. It is a popular domestic diuretic.

OLEUM LAVANDULÆ FLORUM. Oil of Lavender Flowers.

A volatile oil distilled from the fresh flowering tops of *Lavandula officinalis* Chaix (Fam. *Labiatae*). It should be kept in amber-colored, well-stoppered bottles, in a cool place, protected from light.

A colorless or yellow liquid, having the fragrant odor of lavender flowers, and a pungent and slightly bitter taste.

Specific gravity: 0.880 to 0.892 at 25° C. (77° F.).

It is soluble in 3 parts of 70 per cent. alcohol.

When the oil is shaken with water in a narrow graduated cylinder, its volume should not be diminished (absence of *alcohol*).

Tests for Identity.—Given above.

Impurities.—Alcohol. Details above.

Composition.—Lævogyrate linalyl acetate, 30 to 45 per cent.; geraniol, some terpenes, etc.

Remarks.—*Oil of spike* is a crude oil of lavender, made by distilling the lavender herb.

Medical Properties.—Stimulant. Used mostly as perfume. Official preparations: *Spirit of lavender* (p. 180) and *compound tincture of lavender* (p. 230). A constituent of *aromatic spirit of ammonia* (p. 490).

Dose.—0.2 Cc. (3 minims).

OLEUM ROSÆ. Oil of Rose.

A volatile oil distilled from the fresh flowers of *Rosa damascena* Mueller (Fam. *Rosaceæ*), having, when assayed by the process given on p. 1043, a saponification value of not less than 10 nor more than 17. It should be kept in well-stoppered, amber-colored vials, in a cool place, protected from light. When dispensed, it should be completely liquefied by warming, if necessary, and well mixed by agitation.

A pale yellowish, transparent liquid, having the strong, fragrant odor of rose, and a mild, slightly sweetish taste.

Specific gravity: 0.855 to 0.865 at 25° C. (77° F.).

The addition of 70 per cent. alcohol precipitates the paraffin hydrocarbons of the Oil, but forms a clear solution with its other constituents, the solution being slightly acid to litmus T.S.

The congealing point, when determined according to the following method, should be between 18° and 22° C. (64.4° and 71.6° F.).

Introduce about 10 Cc. of Oil into a test-tube of about 15-Mm. diameter; insert a thermometer in such a manner that it touches neither the bottom nor the sides of the tube. Raise the temperature of the Oil in the tube from 4° to 5° above the saturation point by grasping it in the hand, and shake the tube gently. Allow the Oil to cool, and when the first crystals appear, note the temperature. This is regarded as the congealing point; a second test should be made for confirmation.

Composition.—Liquid portion consists of geraniol (p. 810), 70 per cent.; citronellol, 20 per cent. (p. 810); esters, 2.5 to 3.5 per cent. Stearopten is a mixture of paraffin hydrocarbons.

Remarks.—As shown in the official description, oil of rose consists of an eleoptene which contains the odorous principles, and an odorless stearoptene. The pure oil is very expensive, since it requires 3000 kilos of fresh petals to yield one kilo of oil; hence the commercial article is grossly adulterated—at place of distillation in Bulgaria—with palmarosa oil (Turkish geranium oil), and in the open market with a host of other substances.

As these adulterants contain no stearopten, the congealing point test of the pharmacopœia is aimed to distinguish the pure oil from adulterated specimens, but as the spermaceti is added to sophisticated oils to make up for the lack of the true stearopten, the test loses some of its value. As palmarosa oil consists largely of geraniol, assays for that body have no value in determining purity of rose oil; hence chemists rely chiefly on the saponification figures as outlined in the official definition given above. The saponification assay will be left for consideration under volumetric analysis (p. 1043), so here it will be mentioned that it is the esters of the oil which are saponified (see p. 737), and that the amount of these is much smaller than the amount found in palmarosa oil. Thus, while the saponification number of oil of rose is 10 to 17, that of palmarosa oil is 30 to 50.

Medical Properties.—Used as a perfume.

OLEUM ROSMARINI. Oil of Rosemary.

A volatile oil distilled from the fresh flowering tops of *Rosmarinus officinalis* Linné (Fam. *Labiatae*), yielding, when assayed by the process given on p. 1044, not less than 2.5 per cent. of ester, calculated as bornyl acetate, and not less than 10 per cent. of total borneol. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow, limpid liquid, having the characteristic, pungent odor of rosemary, and a warm, somewhat camphoraceous taste.

Specific gravity: 0.894 to 0.912 at 25° C. (77° F.).

The Oil should be dextrogyrate, the angle of rotation being not more than +15° in a 100-Mm. tube, at a temperature of 25° C. (77° F.). The first 10 per cent. obtained by a fractional distillation should also be dextrogyrate.

Soluble in about one-half volume or more of 90 per cent. alcohol; also in 2 to 10 volumes of 80 per cent. alcohol.

Tests for Identity.—Given above.

Assay.—Details in Part V.

Composition.—Pinene, camphene, cineol, camphor, borneol, esters, etc.

Remarks.—It will be seen from the official definition that the official oil must contain 2.5 per cent. of ester and 10 per cent. borneol. The ester estimation is by saponification number, while the borneol is assayed by means of the acetylation test. These latter, like the saponification test, will be explained in chapter on Volumetric Analysis (p. 1044).

Medical Properties.—Carminative.

Dose.—0.2 Cc. (3 minims).

OLEUM SANTALI. Oil of Santal.

A volatile oil distilled from the wood of *Santalum album* Linné (Fam. *Santalaceae*), yielding, when assayed by the process given on p. 1044, not less than 90 per cent. of alcohols, calculated as santalol. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A pale yellow, somewhat thick liquid, having a peculiar, aromatic odor, and a pungent, spicy taste.

Specific gravity: 0.965 to 0.975 at 25° C. (77° F.).

The Oil is lævogyrate; its angle of rotation should be not less than -16° nor more than -20° in a 100-Mm. tube, at a temperature of 25° C. (77° F.) (absence of other varieties of sandalwood oil, etc.).

Readily soluble in alcohol, the solution being slightly acid to blue litmus-paper. It is soluble in 5 volumes of 70 per cent. alcohol.

Tests for Identity.—Given above.

Impurities.—Other sandalwood oils. Details above. Chlorinated products. Details under Benzaldehyde.

Assay for Santalol.—Details in Part V.

Composition.—Santalol, $C_{15}H_{25}OH$; santalal, $C_{15}H_{24}O$, an aldehyde; esters.

Remarks.—It will be noted that this oil comes from a different source than the official red saunders.

Medical Properties.—Antiseptic in gonorrhea.

Dose.—0.5 Cc. (8 minims).

OLEUM THYMI. Oil of Thyme.

A volatile oil distilled from the leaves and flowering tops of *Thymus vulgaris* Linné (Fam. *Labiatae*), and containing, when assayed by the process given below,

not less than 20 per cent., by volume, of phenols. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless liquid, having a strong odor of thyme, and an aromatic, pungent, afterward cooling taste.

Specific gravity: 0.900 to 0.930 at 25° C. (77° F.).

It is slightly levogyrate; not more than -3° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Oil of Thyme is soluble in half its volume of alcohol, also in 1 to 2 volumes of 80 per cent. alcohol.

With a drop of ferric chloride T.S. it yields a greenish-brown color, which changes to reddish.

If 1 Cc. of Oil of Thyme be shaken with 10 Cc. of hot water, and, after cooling, the liquid be passed through a wet filter, the filtrate should not assume, with a drop of ferric chloride T.S., a bluish or violet color (absence of *official phenol*).

ASSAY

Introduce 20 Cc. of solution of sodium hydroxide (1 in 20) into a burette of 50-Cc. capacity (graduated in tenths). Add 10 Cc. of the Oil to be assayed, stopper the burette with a well-fitting cork, shake the mixture thoroughly, and set aside for twelve to twenty-four hours. Drops of Oil adhering to the side of the burette should be loosened by tapping and rotating the burette. After the alkaline solution has become clear, the volume of non-phenol oil remaining (which should measure not more than 8 Cc.) is noted and subtracted from the 10 Cc. of the Oil originally taken. The difference, multiplied by 10, indicates the percentage of phenols in the Oil.

Tests for Identity.—Given above.

Impurity.—Phenol. Details above.

Assay.—Details above.

Composition.—Thymol, cumene, levogyrate pinene, borneol, linalol, carvacrol, etc.

Remarks.—The valuation of oil of thyme is based by the U. S. P. VIII. on the phenol content, which should be at least 20 per cent. The assay given above depends on the fact that the phenols, when treated with alkali, saponify and become soluble in water. Thus, from what was originally 10 Cc. of water-insoluble oil, 2 Cc. become soluble through action of alkali, leaving 8 Cc. of water-insoluble residue.

Medical Properties.—Carminative and antiseptic. Used in *compound antiseptic solution*, U. S. P. (p. 184).

Dose.—0.2 Cc. (3 minims).

VOLATILE OIL DRUGS

VALERIANA. Valerian.

The dried rhizome and roots of *Valeriana officinalis* Linné (Fam. *Valerianaceæ*).

Constituents.—Volatile oil, resin, acids, etc.

The volatile oil is composed of the terpenes, camphene and pinene; borneol esters, chiefly the valerianate, 9.5 per cent., and several other bodies. It is now thought that all the valerianic acid present is in the form of the ester above mentioned.

Medical Properties.—Sedative in hysteria. Administered as *fluid-extract*, U. S. P. (p. 246), *tincture*, U. S. P. (p. 237), and the *ammoniated tincture*, U. S. P. (p. 237). The valerianates, especially ammonium valerianate, are popular, the unofficial elixir of the latter being largely employed.

Dose.—Of drug, 2 Gm. (30 grains).

VIBURNUM OPULUS. *Viburnum Opulus.* Cramp Bark.

The dried bark of *Viburnum Opulus* Linné (Fam. *Caprifoliaceæ*).

VIBURNUM PRUNIFOLIUM. *Viburnum Prunifolium.* Black Haw.

The dried bark of the root of *Viburnum prunifolium* Linné, or of *Viburnum Lentago* Linné (Fam. *Caprifoliaceæ*).

Constituents.—Both contain valerianic acid and bitter, resinous principles, one of the latter found in both drugs called *viburnin*.

Remarks.—These two barks are placed in this chapter merely for the lack of a better position. If they possess any medicinal activity, it is due to the valerianic acid they contain, hence they can best be considered along with valerian. Bear in mind, however, that *neither drug contains volatile oil*, hence their study in this chapter is, to a certain extent, inconsistent and possibly misleading.

Medical Properties.—Both drugs are popularly esteemed as nervines, especially in internal disorders; having been of late largely advertised among physicians, through the exploiting of a proprietary prescription. A similar preparation is found in the National Formulary—Compound Elixir of *Viburnum Opulus*, N. F. The present pharmacopœia recognizes the *fluidextract* (p. 247) of each drug.

Dose.—Of each drug, 2 Gm. (30 grains).

SASSAFRAS. *Sassafras.*

The dried bark of the root of *Sassafras variifolium* (Salisbury) O. Kuntze (Syn. *Sassafras Sassafras* (Linné) Karsten) (Fam. *Lauraceæ*), collected in early spring or autumn, and deprived of the periderm.

Constituents.—Volatile oil, 7 per cent.; resin, a tannin-like body called *sassafrid*, tannic acid, etc.

Medical Properties.—Carminative, diaphoretic (as hot infusion).

Dose.—8 Gm. (120 grains).

OLEUM SASSAFRAS. Oil of Sassafras.

A volatile oil distilled from the root, especially the root bark, of *Sassafras variifolium* (Fam. *Lauraceæ*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A yellow or reddish-yellow liquid, having the characteristic odor of sassafras, and a warm, aromatic taste.

Specific gravity: 1.065 to 1.075 at 25° C. (77° F.).

The Oil is dextrogyrate, but should not deviate the ray of polarized light more than +4° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Soluble in all proportions in 90 per cent. alcohol, the solution being neutral to litmus-paper.

Composition.—Safrol, 80 per cent.; pinene and phellandrene, 10 per cent.; dextrogyrate camphor, 6.8 per cent.; eugenol, sesquiterpenes, etc.

Remarks.—Note that safrol is recognized by the present pharmacopœia (see p. 798).

Medical Properties.—Stimulant and carminative. Used largely in liniments.

Dose.—0.2 Cc. (3 minims).

CINNAMOMUM SAIGONICUM. Saigon Cinnamon.

The bark of an undetermined species of *Cinnamomum* (Fam. *Lauraceæ*).

CINNAMOMUM ZEYLANICUM. Ceylon Cinnamon.

The inner bark of the shoots of *Cinnamomum zeylanicum* Breyne (Fam. *Lauraceæ*)

Constituents.—Both barks contain volatile oil, mucilage, only traces of starch, etc.

Remarks.—These two barks are similar in properties, but widely different in appearance, Saigon cinnamon being in pieces not unlike ordinary barks of same sizes; while Ceylon cinnamon, which is the inner bark, is as thin as paper, and comes in tightly rolled quills. The oil from these two barks is not identical with the official oil.

Official preparations (all from the Saigon bark): *Tincture cinnamon*, *compound tincture of cardamom*, *compound tincture of gambir*, *compound tincture of lavender*, *aromatic tincture of rhubarb*, and *aromatic powder*.

Medical Properties.—Carminative, flavor.

Dose.—Of each bark, 250 milligrammes (4 grains).

Cassia cinnamon, the bark of *Cinnamomum Cassia*, was recognized by the pharmacopœia of 1890, but was dropped in the last revision.

OLEUM CINNAMOMI. Oil of Cinnamon. Oil of Cassia.

A volatile oil distilled from Cassia Cinnamon (Fam. *Lauraceæ*), yielding, when assayed by the process given below, not less than 75 per cent., by volume, of cinnamic aldehyde. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A yellowish or brownish liquid, becoming darker and thicker by age and exposure to the air, having the characteristic odor of cinnamon, and a sweetish, spicy, and burning taste.

Specific gravity: 1.045 to 1.055 at 25° C. (77° F.). The Oil (or if it be dark, its distillate) should be optically almost inactive; it should not be more than one degree dextrogyrate or levogyrate when viewed through a 100-Mm. tube.

Soluble in 2 volumes of 70 per cent. alcohol.

When shaken with a saturated solution of sodium bisulphite, it solidifies to a crystalline mass.

If 4 drops of the Oil, contained in a test-tube, be cooled to 0° C. (32° F.), and then shaken with 4 drops of fuming nitric acid, crystalline needles or plates will be formed.

If a portion of the oil be shaken with hydrogen sulphide T.S., it should not assume a dark color (absence of *lead* and *copper*).

If 1 Cc. of the Oil be mixed with 3 Cc. of a mixture of 3 volumes of alcohol and 1 volume of water, a clear solution should result; and if to this solution there be gradually added 2 Cc. of a saturated solution of lead acetate in a mixture of 3 volumes of alcohol and 1 volume of water, no precipitate should be produced (absence of *petroleum* and of *rosin*).

ASSAY FOR CINNAMIC ALDEHYDE

Introduce into a flask with a long graduated neck (cassia-flask), by means of a measuring pipette, 10 Cc. of the Oil of Cinnamon, add 10 Cc. of a 30 per cent. solution of sodium bisulphite, shake the flask, and heat it in a water-bath containing boiling water until the contents are liquefied; add successive portions (10 Cc. each) of the bisulphite solution, shaking and heating as before, after each addition, until the flask is three-fourths filled. Continue to heat it in the water-bath until the odor of cinnamic aldehyde is no longer perceptible, cool the flask to about 25° C. (77° F.), and add enough of the bisulphite solution to raise the lower limit of the oily layer to the zero mark of the scale. The residual liquid should not measure more than 2.5 Cc., corresponding to at least 75 per cent., by volume, of cinnamic aldehyde.

Tests for Identity.—Given above.

Impurities.—Lead, copper, petroleum, rosin. Details above.

Assay.—Details above.

Composition.—Cinnamic aldehyde, 75 per cent.; methyl-orthocumaric aldehyde, cinnamyl acetate, free cinnamic acid, etc.

Remarks.—It will be noted that the official definition given above does not specify which part of the *Cassia cinnamon* plant furnishes the oil. In truth, all parts yield it, but the commercial source is usually the leaves and twigs. The oil is not identical with that from *Ceylon cinnamon*; in fact, the only point of similarity is that the bulk of both oils consists of cinnamic aldehyde. Note that this body is now official (p. 796). Cinnamic acid present in the oil attacks metals, and as the oil is shipped from China in lead containers, that metal is apt to be found in the oil; hence the pharmacopœial test for lead and copper given above.

The pharmacopœial assay of the oil given above is based on the solubility of cinnamic aldehyde in sodium bisulphite solution, and the method of procedure is, therefore, similar to the assay of the oil of thyme (p. 822). In the present assay, when 10 Cc. of the oil is warmed with the bisulphite solution, all the cinnamic aldehyde goes into solution, leaving the other constituents of the oil floating on top as an oily layer. As the aldehyde should constitute 75 per cent. of the bulk of the oil, the oily residue from 10 Cc. of oil should measure not more than 2.5 Cc. The operation is carried on in a flask with a graduated neck (*Cassia flask*).

Medical Properties.—Stimulant, rubefacient, flavor. Used in preparing *cinnamon water*, U. S. P. (p. 179), and *spirit of cinnamon*, U. S. P. (p. 173).

Dose.—0.05 Cc. (1 minim).

EUCALYPTUS, *Eucalyptus*.

The dried leaves of *Eucalyptus Globulus* Labillardière (Fam. *Myrtaceæ*), collected from the older parts of the tree.

Constituents.—Volatile oil, 6 per cent.; chlorophyll, resin, tannin, etc.

Medical Properties.—Stimulant, carminative, antiseptic. The *fluidextract* (p. 246) is official.

Dose.—2 Gm. (30 grains).

OLEUM EUCALYPTI. Oil of *Eucalyptus*.

A volatile oil distilled from the fresh leaves of *Eucalyptus*, rectified by steam distillation, and yielding, when assayed by the process given below, not less than 50 per cent., by volume, of cineol (*eucalyptol*). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow liquid, having a characteristic, aromatic, somewhat camphoraceous odor, and a pungent, spicy, and cooling taste.

Specific gravity: 0.905 to 0.925 at 25° C. (77° F.).

Soluble in all proportions in alcohol; also soluble in 3 volumes of 70 per cent. alcohol.

Its alcoholic solution should be neutral to litmus-paper.

It is dextrogyrate, the angle of rotation being not more than +10° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

If 2 Cc. of the Oil be mixed with 4 Cc. of glacial acetic acid, and 3 Cc. of a saturated aqueous solution of sodium nitrite be gradually added, the mixture, when gently stirred, should not form crystals of phellandrene nitrite (absence of *eucalyptus* oils containing much *phellandrene*).

ASSAY FOR CINEOL

Introduce into a beaker a solution prepared by dissolving 10 Cc. of Oil of Eucalyptus in 50 Cc. of purified petroleum benzin; immerse the beaker in a freezing mixture and add phosphoric acid, drop by drop, with constant stirring, until the white magma of cineol phosphate formed begins to assume a yellowish or pinkish tint; then transfer the magma to a force filter, wash it with cold purified petroleum benzin, and then dry it by pressure between two porous plates. Transfer the precipitate (cineolphosphate) to a narrow graduated cylinder, and add warm water, which will cause separation of the cineol. The volume, in cubic centimeters, of the separated oil, multiplied by 10, represents the volume per cent. of cineol (eucalyptol). This should correspond to the properties and tests given under *Eucalyptol*.

Tests for Identity.—Given above.

Impurities.—Other eucalyptus oils, with much phellandrene. Details above.

Assay for Cineol.—See above.

Composition.—Eucalyptol (or cineol), 50 per cent.; dextrogyrate pinene; other terpenes (little or no phellandrene), etc.

Remarks.—The cineol assay given above is based on the formation of solid cineol phosphate, which precipitates from benzin solution of the oil. The washed and dried precipitate is then transferred to a graduated cylinder and is treated with warm water, when the cineol separates as an oily liquid, and its volume can be measured. Ten cubic centimeters of oil by this process should yield 5 Cc. of cineol. Note that cineol is official (see Eucalyptol, p. 814).

Medical Properties.—Antiseptic.

Dose.—0.5 Cc. (8 minims).

BUCHU. Buchu.

The dried leaves of *Barosma betulina* (Thunberg) Bartling and Wendland (Fam. Rutaceæ).

Constituents.—Volatile oil, diosmin.

Remarks.—The volatile oil contains a stearopten, *diosphenol* (a phenol, $C_{10}H_{16}O_2$, which turns green black with ferric chloride), while the eleoptene consists of a terpene, $C_{10}H_{18}$, and a ketone resembling menthone.

Remarks.—The most prominent characteristic of this valuable drug of South Africa is its peculiar and pleasant odor.

Medical Properties.—Stimulant, diuretic. Administered as infusion and as *fluidextract*, U. S. P. (p. 246).

Dose.—2 Gm. (30 grains).

HEDEOMA. Hedeoma. Pennyroyal.

The dried leaves and flowering tops of *Hedeoma pulegioides* (Linné) Persoon (Fam. Labiatae).

Constituents.—Volatile oil, etc.

Medical Properties.—Diaphoretic, emmenagogue, carminative.

Dose.—8 Gm. (120 grains).

OLEUM HEDEOMÆ. Oil of Hedeoma.

A volatile oil distilled from the leaves and flowering tops of *Hedeoma*. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light. A pale yellow, limpid liquid, having a characteristic, pungent, mint-like odor and taste.

Specific gravity: 0.920 to 0.935 at 25° C. (77° F.).

It should form a clear solution with 2 volumes or more of 70 per cent. alcohol.

It is dextrogyrate, the angle of rotation varying from about +18° to +22° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Tests for Identity.—Given above.

Composition.—*Pulegone*, $C_{10}H_{16}O$; two other ketones.

Medical Properties.—Emmenagogue. As it is used as an abortifacient, its sale is prohibited in many States.

Dose.—0.2 Cc. (3 minims).

MENTHA PIPERITA. Peppermint.

The dried leaves and flowering tops of *Mentha piperita* Linné (Fam. *Labiata*).

Constituents.—Volatile oil, tannic acid, etc.

Medical Properties.—Carminative, stimulant, flavor. The herb is used to color the official *spirit of peppermint*.

Dose.—4 Gm. (60 grains).

OLEUM MENTHÆ PIPERITÆ. Oil of Peppermint.

A volatile oil distilled from the fresh or partly dried leaves and flowering tops of Peppermint, rectified by steam distillation, and yielding, when assayed by the process given on p. 1043, not less than 6 per cent. of ester, calculated as menthyl acetate, and not less than 50 per cent. of total menthol (free and as ester). It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless liquid, having the characteristic strong odor of peppermint, and a strongly aromatic, pungent taste, followed by a sensation of cold when air is drawn into the mouth.

Specific gravity: 0.894 to 0.914 at 25° C. (77° F.).

It forms a clear solution, neutral to litmus-paper, with an equal volume of alcohol; also soluble in 4 volumes of 70 per cent. alcohol, the solution showing not more than a slight opalescence.

It is levogyrate, the angle of rotation varying from -25° to -33° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

If from 25 Cc. of Oil about 1 Cc. be distilled and the distillate poured on an aqueous solution of mercuric chloride, a white film should not form at the zone of contact after a short time (absence of *dimethyl sulphide* found in non-rectified oils).

Tests for Identity.—Given above.

Impurities.—Dimethyl sulphide. Details above.

Assay.—Details in Part V.

Composition.—Menthol, U. S. P.; methyl acetate; menthane and fourteen other bodies.

Remarks.—The different peppermint oils—American, English, Japanese, German—are not identical in chemical composition, the statement just given referring to the American oil. Thus the Japanese oil contains 70-91 per cent. total menthol, the English 58 to 66 per cent., while the American has 50 to 60 per cent. The menthol content was formerly considered the criterion of quality of the oil; hence the test provided in the former pharmacopœias that a sample of oil placed in freezing mixture should yield crystals of menthol. Experience has

shown, however, that oil of the best quality did not respond to this test, which was, therefore, omitted in the last revision. The assay for menthol (acetylation test) and of methyl acetate (saponification test) will be given under volumetric analysis.

Medical Properties.—Carminative and flavor. Administered as *spirit*, U. S. P. (p. 180). The oil is a constituent of *peppermint water*, U. S. P. (p. 174) and of peppermint lozenges, U. S. P. 1890.

Dose.—0.2 Cc. (3 minims).

MENTHA VIRIDIS. Spearmint.

The dried leaves and flowering tops of *Mentha spicata* Linné (*Mentha viridis* Linné) (Fam. *Labiata*).

Constituents.—Volatile oil, resin, etc.

Medical Properties.—Similar to peppermint. Same dose.

OLEUM MENTHÆ VIRIDIS. Oil of Spearmint.

A volatile oil distilled from the fresh or partly dried leaves and flowering tops of Spearmint, rectified by steam distillation. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless, yellow, or greenish-yellow liquid, having the characteristic, strong odor of spearmint, and a hot, aromatic taste.

Specific gravity: 0.914 to 0.934 at 25° C. (77° F.).

With an equal volume of 80 per cent. alcohol it forms a clear solution, which upon further dilution becomes turbid.

It is levogyrate, the angle of rotation varying from -35° to -48° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

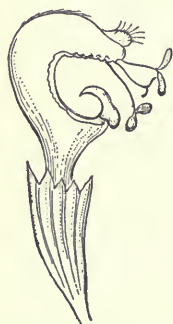


Fig. 226.—Horse-mint flower.

Composition.—Lævogyre carvone, 56 per cent. terpenes (probably l-limonene and l-pinene).

Medical Properties.—Like oil of peppermint and in same dose.

Spirit of Spearmint, U. S. P. (p. 180), is made from the oil and a small amount of the herb.

All three drugs, hedeoma, peppermint, and spearmint, are representatives of the *Labiata*, the "lip family," or mint family; this group of plants being so called because the flowers of this family have their petals united with a two-lipped corolla, as shown in (Fig. 226), the flower of the horsemint.

The plants of this family are characterized by the presence of aromatic volatile oils, and a large number of them are used in medicine. Among those formerly official may be cited *melissa* (Balm), *catnip*, *thyme*, and *origanum*, while three others, scullcap, horehound, and boneset, are still official and will be considered in this chapter.

CARYOPHYLLUS. Cloves.

The dried flower buds of *Eugenia aromatica* (Linné) O. Kuntze (Fam. *Myrtaceæ*).

Constituents.—Volatile oil, tannin, gum resin, fixed oil.

Remarks.—Cloves are not fruit nor seed, as is commonly believed, but are flower-buds. Sliced lengthwise, the sample is found hollow

in the upper parts, where will be found the stamens, which look like dried threads. Cloves are used as a spice, and are largely adulterated. For instance, whole cloves from which the oil has been distilled have been put on the market. These are detected by the fact that such exhausted cloves float horizontally on the surface of water, while the true article, containing the heavy volatile oil, will either sink or float in an upright position. The powder is adulterated with starchy material or with mineral substances, which impurities can be detected by the fact that pure powdered cloves, when viewed under the microscope, contain few or no starch-grains, no "stone-cells" (as does ground cocoanut shell, another adulterant), and yield, on burning, no more than 8 per cent. of ash.

Cloves are used in the following official preparations: *Compound tincture of lavender* and *aromatic tincture of rhubarb*.

Dose.—250 milligrammes (4 grains).

OLEUM CARYOPHYLLI. Oil of Cloves.

A volatile oil distilled from Cloves, yielding, when assayed by the process given below, not less than 80 per cent., by volume, of eugenol. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow, thin liquid, becoming darker and thicker by age and exposure to the air, having a strongly aromatic odor of cloves, and a pungent and spicy taste.

Specific gravity: 1.040 to 1.060 at 25° C. (77° F.).

Soluble in an equal volume of alcohol, this solution being slightly acid to litmus-paper; also soluble in about 2 volumes of 70 per cent. alcohol.

When shaken with an equal volume of a concentrated solution of potassium hydroxide, or of stronger ammonia water, it forms a semisolid, yellowish mass.

If 2 drops of the Oil be dissolved in 4 Cc. of alcohol, and a drop of ferric chloride T.S. added, a bright green color will be produced; and if the same test be made with a drop of diluted ferric chloride T.S., prepared by diluting the test solution with four times its volume of water, a blue color will be produced, which soon changes to yellow.

ASSAY FOR EUGENOL

Introduce into a flask with a long neck (graduated in tenths) 10 Cc. of the Oil of Cloves and 100 Cc. of potassium hydroxide T.S., and shake the mixture for five minutes. When the liquids have separated completely, add sufficient potassium hydroxide T.S. to raise the lower limit of the oily layer to the zero mark of the scale, and note the volume of the residual liquid, which should not measure more than 2 Cc., indicating the presence of at least 80 per cent. of eugenol.

Tests for Identity.—Given above.

Impurities.—Phenol. Details under Eugenol (p. 797).

Assay for Eugenol.—Details above.

Composition.—Eugenol, 70 to 85 per cent.; acetoeugenol, 2 to 3 per cent.; caryophyllene, $C_{15}H_{24}$, etc.

Remarks.—The assay given above is based on the same principle as that of oil of thyme (p. 822), namely, dissolving of the phenol in alkaline solution and measuring the residual liquid.

Medical Properties.—Stimulant. Used in toothache.

Dose.—0.2 Cc. (3 minims).

ANISUM. Anise.

The ripe fruit of *Pimpinella Anisum* Linné (Fam. *Umbelliferae*), obtained from cultivated plants.

Constituents.—Volatile oil, fixed oil.

Remarks.—The pharmacopœia directs that anise, when triturated with potassium hydroxide solution, should not give a mouse-like odor. This test is to distinguish it from the poisonous conium (p. 926), which closely resembles anise in appearance.

Medical Properties.—Carminative, stimulant, flavor.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Illicium, or *star anise*, the fruit of *Illicium verum*, was official in the pharmacopœia of 1890, and is here mentioned because it yields an oil identical with that from anise.

OLEUM ANISI. Oil of Anise.

A volatile oil distilled from Anise or from the fruit of Star Anise, *Illicium verum* Hooker filius (Fam. *Magnoliaceæ*). It should be kept in well-stoppered, amber-colored bottles, protected from light, and, if it has separated into a liquid and a solid portion, it should be completely liquefied by warming, and then well shaken, before being dispensed.

A colorless or pale yellow, thin, and strongly refractive liquid, having the characteristic odor of anise, and a sweetish, mildly aromatic taste.

Specific gravity: 0.975 to 0.985 at 25° C. (77° F.).

The Oil should be levogyrate, the angle of rotation being -2° in a 100-Mm. tube, at a temperature of 25° C. (77° F.) (absence of *oil of fennel*, etc.).

Soluble in an equal volume of alcohol, forming a clear solution, also in 5 volumes of 90 per cent. alcohol (absence of *petroleum*, of *most fixed oils*, and of *oil of turpentine*).

An alcoholic solution of Oil of Anise is neutral to litmus-paper, and should not assume a blue or brownish color on the addition of a drop of ferric chloride T.S. (absence of some *volatile oils* containing *phenols*).

When the Oil is shaken with water in a narrow graduated cylinder, its volume should not be diminished (absence of *alcohol*).

When tested according to the following method, the congealing point of Oil of Anise should not be below 15° C. (59° F.).

Transfer about 10 Cc. of the Oil to a test-tube, placed in water cooled with ice; insert a thermometer at once into the Oil, and allow it to remain undisturbed until its temperature has fallen to about 6° C. (42.8° F.). Induce crystallization either by rubbing the inner wall of the test-tube with the thermometer, or by the addition of a particle of solid anethol, remove the test-tube from the bath, and stir continuously during the solidification of the Oil.

The highest temperature reached during the crystallization is regarded as the congealing point.

Tests for Identity.—Given above.

Impurities.—Oil of fennel, petroleum, fixed oils, oil of turpentine, oil containing phenols, alcohol. Details above.

Congeeing Point.—At least 15° C. Details above.

Composition.—Anethol, 80 to 90 per cent.; methyl-chavicol, both $C_{10}H_{12}O$.

Remarks.—As anethol is a solid, the congealing point of the oil (15° C.) is a criterion of value.

Medical Properties.—Carminative and flavor. Used in making *anise water* (p. 171) and *spirit of anise* (p. 178).

Dose.—0.2 Cc. (3 minims).

CORIANDRUM. Coriander.

The dried ripe fruit of *Coriandrum sativum* Linné (Fam. *Umbelliferae*).

Constituents.—Volatile oil, $\frac{1}{2}$ to 1 per cent. fixed oil, etc.

Medical Properties.—Aromatic, carminative.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

OLEUM CORIANDRI. Oil of Coriander.

A volatile oil distilled from Coriander. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or slightly yellow liquid, having the characteristic, aromatic odor of coriander, and a warm, spicy taste.

Specific gravity: 0.863 to 0.878 at 25° C. (77° F.).

It should be soluble in 3 volumes of 70 per cent. alcohol; also soluble in all proportions in 80 per cent. and 90 per cent. alcohol.

It is dextrogyrate, the angle of rotation varying from +7° to +14° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Composition.—Dextrogyrate linalool; pinene, etc.

Medical Properties.—Carminative.

Dose.—0.2 Cc. (3 minims).

FÆNICULUM. Fennel.

The dried, nearly ripe fruit of *Feniculum vulgare* Miller (Fam. *Umbelliferae*).

Constituents.—Volatile oil, 2 to 6 per cent.; fixed oil, etc.

Medical Properties.—Carminative, stimulant.

Dose.—1 Gm. (15 grains).

OLEUM FÆNICULI. Oil of Fennel.

A volatile oil distilled from Fennel. It should be kept in well-stoppered, amber-colored bottles, in a cool place, and, if it has partly or wholly solidified, it should be completely liquefied by warming, and then well shaken before being dispensed.

A colorless or pale yellow liquid, having the characteristic, aromatic odor of fennel, and a sweetish, mild, and spicy taste.

Specific gravity: 0.953 to 0.973 at 25° C. (77° F.).

Soluble in an equal volume of alcohol, the solution being neutral to litmus-paper; also soluble in 10 volumes or less of 80 per cent. alcohol.

Tests for Identity.—Given above.

Impurities.—Oils containing phenols. Test as given under Oil of Anise.

Congealing Point.—At least 5° C. Details as given under Oil of Anise, except that oil is to be chilled to -5° C.

Composition.—*Anethol*, 50 to 60 per cent.; *fenchone*, a ketone having formula $C_{10}H_{16}O$; pinene; dipentene; etc.

Medical Properties.—Carminative. Very popular among German physicians as a constituent of digestive powders. It is a constituent of *compound licorice powder* (p. 303) and of *compound spirit of juniper* (p. 179).

Dose.—0.2 Cc. (3 minims).

CARUM. Caraway.

The dried fruit of *Carum Carvi* Linné (Fam. *Umbelliferae*).

Constituents.—Volatile oil, 5 per cent.; fixed oil; resin.

Medical Properties.—Carminative, stimulant, flavor.

Dose.—1 Gm. (15 grains).

OLEUM CARI. Oil of Caraway.

A volatile oil distilled from Caraway and rectified by steam distillation. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow, thin liquid, having the characteristic, aromatic odor of caraway, and a spicy taste.

Specific gravity: 0.905 to 0.915 at 25° C. (77° F.).

Soluble in an equal volume of alcohol; also in from 3 to 10 volumes of 80 per cent. alcohol.

Oil of Caraway is dextrogyrate, the angle of rotation varying from +70° to +80° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Tests for Identity.—Given above.

Composition.—Carvone, 50 to 60 per cent.; dextrogyrate limonene; etc.

Medical Properties.—Carminative. A constituent of *compound spirit of juniper* (p. 179).

Dose.—0.2 Cc. (3 minims).

The drugs last studied—*anise*, *fennel*, *coriander*, and *caraway*—are representatives of the *Umbelliferae*; the *parsley* family, whose flowers are arranged on stalks which radiate like rods of an umbrella, as shown in Fig. 227.

Most official drugs yielded by the *Umbelliferae* are fruit, which are, unfortunately, commonly called “seeds.”



Fig. 227.—Inflorescence of umbellifera (schematic).

A fruit can vary from a seed, with its dry ovary wall, to very elaborate fruit, which include several other organs attached to the ovary. Thus, when we examine an apple, we find three distinct layers—the red skin, the white meat, and the papery core surrounding the seed. Of these layers, only the core represents the ovary wall, the meat and skin being a modification of another part of the flower—the green calyx. On the other hand, an acorn likewise represents a true fruit, the cup being a cluster of leaves at the base of the ripened ovary, the latter being the “nut.” From this can be separated the outer coat, which represents the ovary wall, while the “kernel” within, with its shaggy coat, represents the seed. Similarly, the so-called “seeds”—*anise*, *caraway*, *coriander*, and *fennel*—are fruit just as are apples and acorns, and, like the acorn, the ovary wall is inconsiderable, and does not separate from the seed of its own free will.

PIMENTA. *Pimenta*. Allspice.

The dried, nearly ripe fruit of *Pimenta officinalis* Lindley (Fam. *Myrtaceae*).

Constituents.—Volatile oil, resin, fixed oil, chlorophyll. Traces of alkaloid.

Medical Properties.—Stomachic, carminative.

Dose.—1 Gm. (15 grains).

OLEUM PIMENTÆ. Oil of *Pimenta*. Oil of Allspice.

A volatile oil distilled from *Pimenta*, yielding, when assayed by the process given below, not less than 65 per cent., by volume, of eugenol. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless, yellow, or reddish liquid, having a strong, aromatic odor of allspice, and a pungent, spicy taste.

Specific gravity: 1.033 to 1.048 at 25° C. (77° F.).

With 90 per cent. alcohol it is miscible in all proportions. It is also soluble in 2 volumes of 70 per cent. alcohol.

When mixed with an equal volume of a concentrated solution of sodium hydroxide, it forms a semisolid mass.

Tests for Identity.—Given above.

Assay for Eugenol.—Details given under Oil of Cloves.

Constituents.—Eugenol, sequiterpenes, etc.

Remarks.—It will be noticed that the pharmacopœia provides eugenol assay for the oil, identical with that for oil of cloves.

Medical Properties.—Carminative and flavor.

Dose.—0.2 Cc. (3 minims).

CARDAMOMUM. Cardamom.

The dried nearly ripe fruit of *Elettaria repens* (Sonnerat) Baillon (Fam. *Zingiberaceæ*).

Constituents.—Volatile oil, fixed oil, starch, etc. The volatile oil consists of terpenes (dipentane and terpene), terpineol, cineol, terpinyl acetate.

Remarks.—The entire fruit including the pericarp (or “hulls”) is official, although the minute seeds contain the entire activity of the drug. Like all drugs used in spices, the commercial powder is apt to be adulterated with mineral matter, hence the pharmacopœia directs that it should yield not more than 4 per cent. of ash.

Medical Properties.—Carminative, stomachic. Official preparations: The *tincture* (p. 225) and the *compound tincture* (p. 225). Also used in *compound tincture of gentian* (p. 227), *tincture of rhubarb* (p. 235), and *aromatic powder* (p. 301).

Dose.—1 Gm. (15 grains).

SABAL. Sabal.

The partially dried fruit of *Serenoa serrulata* (Roemer and Shultes) Hooker filius (Fam. *Palmeæ*).

Constituents.—Volatile oil (only from fresh fruit), 1.3 per cent.; fixed oil (ethyl ethers of caproic, caprylic, oleic, and other acids), about 1.6 per cent.; sugar (pentoses), 5.4 per cent. The presence of an alkaloid is a subject of dispute.

Remarks.—As apparently the only really active constituent is volatile oil, the use of the partly dried fruit seems of no service. Several large manufacturers prepare the extract from *fresh fruit*.

Medical Properties.—Supposedly tonic and stimulant, especially to the reproductive organs. Reputation chiefly due to advertising by nostrum makers.

Dose.—1 Gm. (15 grains).

AURANTII AMARI CORTEX. Bitter Orange Peel.

The dried rind of the unripe fruit of *Citrus vulgaris* Risso (Fam. *Rutaceæ*).

Constituents.—Volatile oil; *hesperidin*; an acid, $C_{44}H_{28}O_{14}$; resin; *aurantiamarin*, a bitter glucoside. The volatile oil is chemically

identical with that of sweet orange, except that it has a bitter taste, due to the hesperidin and aurantiamarin it contains. This oil is not official.

Medical Properties.—Aromatic bitter. Official preparations, the tincture (p. 222) and the fluidextract (p. 247) in *compound tincture of cinchona* (p. 226) and *compound tincture of gentian* (p. 227).

Dose.—1 Gm. (15 grains).

AURANTII DULCIS CORTEX. Sweet Orange Peel.

The recently separated outer rind of the ripe fruit of *Citrus Aurantium* Linné (Fam. *Rutaceæ*).

Constituents.—Volatile oil; *hesperidin*, $C_{22}H_{26}O_{12}$, a glucoside that hydrolyzes to *hesperetin*, $C_{16}H_{14}O_6$.

Remarks.—Note that the pharmacopœia directs the use of the outer rind—that is, the thin yellow layer must be separated from the thick white layer (the *zest*). This is important in making pharmaceuticals, since the zest contains a body that blackens with iron salts, and the same will be true of preparations made from the peel not freed from the zest.

OLEUM AURANTII CORTICIS. Oil of Orange Peel.

A volatile oil obtained by expression from the fresh peel of the Sweet Orange. It should be kept in small, well-stoppered, amber-colored bottles, in a cool place, so as to avoid, as far as possible, the development of a terebinthinate odor. Oils that have developed such an odor should not be dispensed.

A pale yellow liquid, having the characteristic, aromatic odor of orange, and an aromatic taste.

Specific gravity: 0.842 to 0.846 at 25° C. (77° F.).

Its optical rotation should be dextrogyrate, not less than 95° in a 100-Mm. tube, at a temperature of about 25° C. (77° F.) (absence of *oil of turpentine*, etc.).

When subjected to careful fractional distillation, any Oil coming over below 170° C. (338° F.) should not yield pinene nitrosochloride and nitrosopinene (derived from added oil of turpentine) when tested in the following manner:

Dissolve 5 Cc. of the fraction to be tested in one-half its volume of glacial acetic acid, add 5 Cc. of amyl nitrite, cool thoroughly in a freezing mixture, and add, very gradually, 5 Cc. of a mixture of equal volumes of hydrochloric acid and glacial acetic acid. Collect any crystals which separate from the blue or greenish liquid upon standing, on a force filter, and wash them with a little alcohol. Transfer the crystals to a flask, add 5 Cc. of alcoholic potassium hydroxide T.S., and heat on a water-bath fifteen minutes. Pour the solution into cold water, collect the precipitate, and wash it with cold water. Recrystallize the dried precipitate from alcohol, and determine the melting-point of the crystals. Nitrosopinene melts at 132° C. (269.6° F.), whereas nitrosolimonene or carvoxime (from limonene, one of the normal constituents of Oil of Orange Peel) melts at 72° C. (161.6° F.).

Tests for Identity.—Given above.

Impurities.—Oil of turpentine. Details above.

Composition.—Dextrogyrate limonene, 90 per cent.; *citral*, derivative of anhydrogeraniol (p. 802); citronellal; esters, etc.

Remarks.—As already mentioned (p. 804), the reason why oil of orange and of lemon become terebinthinate is through the conversion of their limonene into dipentene. This change, of course, ruins the quality of the oil, and it is poor pharmacy to use such oils in preparations. Of the several schemes suggested to prevent this decomposi-

tion, the best is the adding of a small amount of glycerin—say, half an ounce to four ounces of oil—and keeping bottle inverted. In this way the mouth of the bottle is “sealed” with a layer of the glycerin, which prevents access of air. When the oil is to be used, the bottle is placed in an upright position for a few minutes, and the oil decanted from the heavier glycerin layer. This, in the writer’s experience, proved more satisfactory than diluting the oil with alcohol. The commercial oil is sometimes adulterated with oil of turpentine, which can be detected by the fact that the angle of rotation of orange oil is $+95^{\circ}$, while that of oil of turpentine is never more than $+25^{\circ}$. The pharmacopœia provides another turpentine test based on the formation of nitroso-limonene crystals from the oil of orange, in place of the nitroso-pinene obtained from oil of turpentine. The test is given, in full, above. A far more subtle adulteration now in use is the addition of the terpenes from oil of lemon after removing the citral, which is used in making the so-called terpeneless oils (p. 805). For such adulteration the citral assay given under Oil of Lemon might be applied.

Medical Properties.—Flavor. *Official preparation, compound spirit of orange* (p. 178).

Dose.—0.2 Cc. (3 minims).

LIMONIS CORTEX. Lemon Peel.

The recently separated outer rind of the ripe fruit of *Citrus Limonum* Risso (Fam. Rutaceæ).

Constituents.—Volatile oil; hesperidin, etc.

Remarks.—It will be noted that this drug, like orange peel, is the “outer rind” separated from the zest.

Medical Properties.—Flavor. *Official preparation, tincture of lemon peel* (p. 230).

OLEUM LIMONIS. Oil of Lemon.

A volatile obtained by expression from fresh Lemon Peel, yielding, when assayed by the process given on p. 1042, not less than 4 per cent. of aldehyde, calculated as citral. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A pale yellow, limpid liquid, having the fragrant odor of lemon, and an aromatic, somewhat bitter taste.

Specific gravity: 0.851 to 0.855 at 25° C. (77° F.).

It is dextrogyrate; its optical rotation should not be less than $+60^{\circ}$ in a 100-Mm. tube, at a temperature of 25° C. (77° F.). The angle of rotation of the first 10 per cent. of Oil obtained by fractional distillation should not differ more than 2° from that of the original Oil.

Tests for Identity.—Given above.

Assay for Citral.—Details in Part V.

Composition.—Dextrogyrate limonene, 90 per cent.; phellandrene; citral, 7 to 10 per cent.; citronellal; esters, etc.

Remarks.—It will be noticed that while it is claimed that oil of lemon contains 7 to 10 per cent. citral, the pharmacopœia demands not less than 4 per cent. The pharmacopœial assay will be considered in the chapter on Volumetric Analysis.

Medical Properties.—Flavoring. Used in *aromatic spirit of ammonia*, U. S. P. (p. 178) and *compound spirit of orange*, U. S. P. (p. 178). *Spirit of lemon*, U. S. P. 1890, has been replaced by tincture of lemon peel.

Dose.—Of the oil, 0.2 Cc. (3 minims).

Oil of bergamot, U. S. P. 1890, is obtained by expression of the rind of the bergamot orange, the fruit of *Citrus Bergamia*. Its green color is due to the presence of copper from its container. It is used as a perfume.

Oil of neroli, U. S. P. 1890, is the oil obtained by distillation of orange flowers, and is a by-product in the manufacture of orange flower water. It is used in perfumery.

It will be noted that the three oils—orange, lemon, and bergamot—are obtained by expression (as already described on p. 817) of the rind of closely allied fruits; that this group of fruit—orange, lemon, lime, etc.—forms a subdivision of the plant family, Rutaceæ.

MYRISTICA. Myristica. Nutmeg.

The kernel of the ripe seed of *Myristica fragrans* Houttuyn (Fam. *Myristicaceæ*).

Constituents.—Volatile oil, 2 to 8 per cent.; fat, 25 per cent.; starch, etc. That fat which is obtained by expression of nutmeg between hot plates is called *nutmeg butter* or *oil of mace*. It consists of 70 per cent. glyceryl myristate and 20 per cent. olein.

Medical Properties.—Stimulant, stomachic, flavor. Used in *compound tincture of lavender* (p. 230), *aromatic tincture of rhubarb* (p. 235), and *aromatic powder* (p. 301).

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

OLEUM MYRISTICÆ. Oil of Myristica. Oil of Nutmeg.

A volatile oil distilled from Myristica. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A thin, colorless or pale yellow liquid, having a characteristic odor of nutmeg, and a warm, spicy taste.

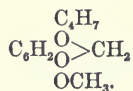
Specific gravity: 0.862 to 0.910 at 25° C. (77° F.).

Soluble in an equal volume of alcohol; also soluble in 3 volumes of 90 per cent. alcohol.

It is dextrogyrate, the angle of rotation varying between +14° and +28° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

When 2 or 3 Cc. of Oil are evaporated on a water-bath, no residue which crystallizes on cooling should be left.

Composition.—Pinene; dipentane; *myristicol*; *myristicin*, etc. Myristicin has the graphic formula:



Medical Properties.—Flavor.

Dose.—0.2 Cc. (3 minims).

Mace (U. S. P. 1890) is the ariloid of the nutmeg. It yields a volatile oil practically identical with that of nutmeg, and, like it, is used as a spice.

An "arilloid," or "arillus," as an outgrowth from the base of the testa or seed coat. The testa of different seeds vary widely in appearance, thus the testa of the almond is not the "shell" of the almond, for the shell is the inner layer of the ovary wall and is a part of the fruit. The almond testa is the thin brown scurfy coating which can be removed from the seed by soaking in warm water.

In other seeds the testa is hard and brittle. Thus cocoanut is a huge seed, and its shell a testa. In myristica the testa is thin and brittle, is loose, and can be easily removed, and the official nutmeg is the kernel of the seed freed from this brittle testa, while mace is the arilloid which branches irregularly over the testa of the unbroken seed.

DRUGS CONTAINING VOLATILE OIL AND RESIN

This class of drugs differs from the volatile oil drugs just considered by the prominence of their resin content. It will be noted that most of the volatile oil drugs just considered contained resinous matter, but these resins were either insignificant as to amount or indifferent as to activity. On the other hand, the drugs now to be considered owe their activity as much to the resin they contain as to their volatile oil, and it will be noted that all the drugs furnishing official pharmaceutic oleoresins (p. 259) are here considered. As to the chemistry of the resinous ingredients of this class of drugs, let it be said that a general consideration of that little understood class of bodies—the resins—is reserved for a special chapter (Chapter XLVIII), which, perhaps, would be the better place to consider drugs of the class under consideration. However, since these drugs are most prominently characterized by their odor, and this is largely due to the volatile oil they contain, it is well to consider them among the volatile oil drugs. It might be stated that all these resinous drugs should be extracted with stronger alcohol.

STILLINGIA. *Stillingia*.

The dried root of *Stillingia sylvatica* Linné (Fam. *Euphorbiaceæ*).

Constituents.—Volatile oil; resin; stillingine, an alkaloid; fixed oil; etc.

Medical Properties.—Nauseant, emetic, in small doses. More or less laxative. *Fluidextract* official (p. 247).

Dose.—2 Gm. (30 grains).

SUMBUL. *Sumbul*. Musk Root.

The dried rhizome and root of an undetermined plant, probably of the family *Umbelliferae*.

Constituents.—Two balsamic resins; trace volatile oil; *angelic acid*, $C_5H_8O_2$; bitter principle, etc.

Medical Properties.—Nerve stimulant. The *extract* (p. 289) and the *fluidextract* (p. 246) are official.

Dose.—2 Gm. (30 grains).

ASPIDIUM. *Aspidium*. Male Fern.

The dried rhizome of *Dryopteris Filix-mas* (Linné) Schott, or of *Dryopteris marginalis* (Linné) Asa Gray (Fam. *Filices*).

Constituents.—Volatile oil; fixed oil; resin; *filicic acid*; *tannaspidic acid*; *pteritannic acid*; *dibutyrilphloroglucin*; *felix red*, etc. The volatile oil consists of the hexyl and octyl esters of the fatty acids (butyric, etc.), also free fatty acids.

Remarks.—Characteristic for good quality of this drug is its green color, which fades as the drug grows old and worthless, hence the pharmacist should use only such samples as are still green.

Medical Properties.—Tapeworm expeller. Used almost entirely in the form of the oleoresin, U. S. P. (p. 261).

Dose.—Of drug, 4 Gm. (60 grains).

ZINGIBER. Ginger.

The dried rhizome of *Zingiber officinale* Roscoe (Fam. *Zingiberaceæ*).

Constituents.—Volatile oil; resin; *gingerol*, etc. The volatile oil consists of a sesquiterpene, dextro-camphene, phellandrene. The resin consists of three distinct resins, and when fused with sodium hydroxide, yields protocathechuic acid.

Remarks.—The best quality of ginger comes from Jamaica, and occurs in handsome lobed pieces coated white with lime. Preserved ginger is the same root collected in the spring when green and succulent and boiled with syrup.

Medical Properties.—Carminative, stimulant, flavor. Official preparations: *Oleoresin* (p. 262), *tincture* (p. 238), and *fluidextract* (p. 246). The last is used in making the *syrup*. Powdered ginger is a constituent of *aromatic powder* (p. 301).

Dose.—Of drug, 1 Gm. (15 grains).

CALAMUS. Calamus. Sweet Flag.

The unpeeled, dried rhizome of *Acorus Calamus* Linné (Fam. *Araceæ*).

Constituents.—Volatile oil; resin; *acorin*; *calamine* (an alkaloid); starch, etc.

The *volatile oil* consists of pinene, a sesquiterpene, and esters. *Acorin* is a bitter glucoside that hydrolyzes to *acoretin* and a resin.

Medical Properties.—Carminative, stimulant, and flavor. The whole peeled root is used for teething infants to chew upon. Note, however, that the unpeeled root constitutes the official drug. The *fluidextract* (p. 246) is official.

Dose.—1 Gm. (15 grains).

CIMICIFUGA. Cimicifuga. Black Snakeroot.

The dried rhizome and roots of *Cimicifuga racemosa* (Linné) Nuttall (Fam. *Ranunculaceæ*).

Constituents.—Volatile oil, resin, tannin, etc.

Medical Properties.—Tonic and "blood purifier" in domestic practice.

Dose.—1 Gm. (15 grains).

SERPENTARIA. *Serpentaria*. Virginia Snakeroot.

The dried rhizome and roots of *Aristolochia Serpentina* Linné (Virginia Serpentaria), or of *Aristolochia reticulata* Nuttall (Texas Serpentaria) (Fam. *Aristolochiaceae*).

Constituents.—Volatile oil; bitter principle, similar to quassin; resin, *aristolochine*.

The volatile oil consists chiefly of borneol, the Texas snakeroot containing also pinene.

Remarks.—It will be observed that official serpentaria is derived from two different plants, the drugs, however, being practically identical in medicinal action.

Medical Properties.—Popular tonic and "blood purifier." The *fluidextract* (p. 246) and the *tincture* (p. 236) are official, while the drug is a constituent of *compound tincture of cinchona*.

Dose.—1 Gm. (15 grains).

XANTHOXYLUM. *Xanthoxylum*. Prickly Ash.

The dried bark of *Xanthoxylum americanum* Miller (Northern Prickly Ash), or of *Fagara Clava-Herculis* (Linné) Small (Southern Prickly Ash) (Fam. *Rutaceae*).

Constituents.—Volatile oil, resin, a bitter principle, *xanthoxylin*, berberin. Xanthoxylin from northern prickly ash is $C_{15}H_{14}O_4$, while that from the southern is $C_{14}H_{12}O_4$.

Remarks.—As in the case of serpentaria, two different plants yield official prickly ash. The constituents of the two barks are practically identical, although it is said that the bitter principle (*xanthoxylin*) from the two is not exactly the same.

The curious name, *xanthoxylum*, has a distinct and sensible meaning, and the best way in which to present the matter forcibly is to mention three drugs between whose names there exists a similarity.

These are:

Xanthoxylum, from the Greek "*xanthos*," yellow, and "*xylon*," wood, or yellow wood.

Erythroxylin, from the Greek "*erythros*," red, and "*xylon*," wood, or red wood.

Hæmatoxylin, from the Greek "*haima*," the blood, and "*xylon*," wood, or blood wood.

Medical Properties.—Alterative, diuretic, sialagogue. Administered as *fluidextract* (p. 246).

Dose.—2 Gm. (30 grains).

MEZEREUM. *Mezereum*.

The dried bark of *Daphne Mezereum* Linné, and of other European species of *Daphne* (Fam. *Thymelaeaceae*).

Constituents.—Acrid resin; trace of acrid volatile oil; daphnin. Daphnin is a glucoside that hydrolyzes to daphnetin.

Medical Properties.—Irritant, sialagogue, externally as a rubefacient. The *fluidextract* (p. 246) is official, and the drug is a constituent of *compound fluidextract of sarsaparilla* (p. 254).

MATICO. Matico.

The leaves of *Piper angustifolium* Ruiz and Pavon (Fam. *Piperaceæ*).

Constituents.—Volatile oil, resin, tannin.

The *volatile oil* consists of methyl-eugenol and asarone.

Medical Properties.—Vulnerary and styptic. Administered as *fluidextract*, U. S. P. (p. 246).

Dose.—4 Gm. (60 grains).

ERIODICTYON. Eriodictyon. Yerba Santa.

The dried leaves of *Eriodictyon californicum* (Hooker and Arnott) Greene (Fam. *Hydrophyllaceæ*).

Constituents.—Resins, trace of volatile oil, tannin, eriodictyol (a phenol), $C_{15}H_{12}O_6$, homo-eridictyol (a phenol, $C_{16}H_{14}O_6$), 3 per cent.

Medical Properties.—Expectorant. Chiefly used for masking taste of quinine, the *aromatic elixir of yerba santa*, N. F., and the *aromatic syrup of yerba santa*, N. F., being generally employed for this purpose. These are prepared from the official *fluidextract* (p. 246).

Dose.—1 Gm. (15 grains).

GRINDELIA. Grindelia.

The dried leaves and flowering tops of *Grindelia robusta* Nuttall, or of *Grindelia squarrosa* (Pursh) Dunal (Fam. *Compositæ*).

Constituents.—Volatile oil, resin, *grindelin*.

Medical Properties.—Recommended for asthma. The official *fluidextract* (p. 246) is also used externally as a lotion in the eruption caused by poison oak.

Dose.—2 Gm. (30 grains).

SABINA. Savin.

The tops of *Juniperus Sabina* Linné (Fam. *Coniferæ*).

Constituents.—Volatile oil, resin, etc.

Remarks.—Savin belongs to the natural order *Coniferæ*,—the pine family,—a very large group of plants which furnishes such important products as turpentine and Canada turpentine. Savin represents leafy tops, which are the ends of the branches, hence the youngest and freshest part of the trees.

Medical Properties.—Irritant, emmenagogue. The *fluidextract* (p. 246) is official.

Dose.—0.5 Gm. ($7\frac{1}{2}$ grains).

OLEUM SABINÆ. Oil of Savin.

A volatile oil distilled from the fresh tops of Savin. It should be kept in well-stoppered and amber-colored bottles, in a cool place, protected from light.

A colorless or yellowish liquid, having a peculiar terebinthinate odor, and a pungent, bitter, and camphoraceous taste.

Specific gravity: 0.903 to 0.923 at 25° C. (77° F.).

The Oil is dextrogyrate, the angle of rotation varying between +40° and +60° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Soluble in about one-half volume or more of 90 per cent. alcohol.

Composition.—*Sabinol*, $C_{10}H_{16}O$ (partly as acetic acid ester); *cadinene*, possibly some terpenes, etc.

Medical Properties.—That of drug.

Dose.—0.05 Cc. (1 minim).

CANNABIS INDICA. Indian Cannabis. Indian Hemp.

The dried flowering tops of the pistillate plants of *Cannabis sativa* Linné (Fam. *Moraceæ*), grown in the East Indies and gathered while the fruits are yet undeveloped, and carrying the whole of their natural resin.

Constituents.—Volatile oil; resin; bitter substance; *cannabinine*, an alkaloid.

The *volatile oil* is said by Personne to contain *cannabene*, $C_{18}H_{20}$, while other writers claim it consists of sesquiterpenes. The *resin* called *cannabin* is a mixture, the active ingredient of which is a red oily mixture called *cannabinol*.

Remarks.—It is of interest that while the plant yielding this drug is identical with the American hemp, the latter plant is of little value in medicine, since it contains only a small amount of resin. Houghton, however, claims that the American hemp is as active as the Indian if only properly prepared.

Medical Properties.—Powerful nervous stimulant, the resinous extract being used in the far East by smoking, under the name of *hashish*. The extract, U. S. P. (p. 280), is largely used to relieve the pain of corns, a favorite recipe for this purpose being *compound salicylated collodion*, N. F. The *tincture* (p. 224) and the *fluidextract* (p. 246) are also official.

Dose.—Of drug, 65 milligrammes (1 grain).

CAPSICUM. Capsicum. Red Pepper. Cayenne Pepper.

The dried, ripe fruit of *Capsicum fastigiatum* Blume (Fam. *Solanaceæ*), deprived of its calyx.

Constituents.—Volatile oil (trace); resin; fat, *capsaicin*. The oleoresin consists of two oily liquids, *capsicin* and *capsicol*, combined with capsaicin and fat. *Capsaicin* is a pungent body, having the formula $C_9H_{14}O_2$.

Remarks.—Capsicum belongs to the natural order *Solanaceæ*, the same family of plants to which belongs the Irish potato, the tomato, and the egg-plant; as well as the poisonous drugs, stramonium, belladonna, and hyoscyamus. The official fruit—the pepper pod—is so well known that extended comment is unnecessary.

Medical Properties.—Stimulant, rubefacient, condiment. The *tincture* (p. 224), *fluidextract* (p. 246), and *oleoresin* (p. 261) are official. From the latter the *plaster* (p. 355) is prepared.

Dose.—Of drug, 65 milligrammes (1 grain).

PIPER. Pepper. Black Pepper.

The dried, unripe fruit of *Piper nigrum* Linné (Fam. *Piperaceæ*).

Constituents.—Volatile oil, resin, *piperine* (p. 879), *chavicin*. The volatile oil consists of either dipentane or phellandrene.

Remarks.—Pepper is what is commonly called black pepper, red pepper being given the distinctive name *capsicum*. Note that pepper is the unripe fruit of *Piper nigrum*, the berry which was picked before fully ripe and shriveled through drying. White pepper represents the same fruit allowed to ripen, after which the fleshy portion is removed by hand, leaving only the seed, with a trace of fruit tissue clinging thereto.

Medical Properties.—Irritant, stimulant, condiment. Usually administered in tonic pills as *oleoresin* (p. 262).

Dose.—500 milligrammes (7½ grains).

CUBEBA. Cubeb.

The dried unripe but fully grown fruit of *Piper Cubeba* Linné filius (Fam. *Piperaceæ*).

Constituents.—Volatile oil 10–18 per cent.; resin; *cubebic acid*; *cubebin*.

Remarks.—This drug is an unripe berry, just as is pepper, and from a closely allied plant, both plants belonging to the same genus.

Medical Properties.—Urinary disinfectant, stimulant.

Official Preparations.—The *oil*, *fluidextract* (p. 246), and *oleoresin* (p. 261) are used in gonorrhea mixtures, while the *lozenges* (p. 332) made from the *oleoresin* are used in bronchitis. The ground drug, smoked as cigarettes, has enjoyed considerable popularity as a remedy for catarrhal troubles.

Dose.—1 Gm. (15 grains).

OLEUM CUBEBAE. Oil of Cubeb.

A volatile oil distilled from Cubeb. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless, pale green, or yellow liquid, having the characteristic odor of cubeb, and a warm, camphoraceous, aromatic taste.

Specific gravity: 0.905 to 0.925 at 25° C. (77° F.).

An alcoholic solution of Oil of Cubeb is neutral to litmus-paper.

It is levogyrate, the angle of rotation varying from –25° to –40° in a 100-Mm. tube, at a temperature of 25° C. (77° F.).

Composition.—Two sesquiterpenes, one of which is cadinene; some terpenes (pinene and dipentane), etc.

Medical Properties.—Used largely in gonorrhea mixtures.

Dose.—0.5 Cc (8 minims).

HUMULUS. Hops.

The carefully dried strobiles of *Humulus Lupulus* Linné (Fam. *Moraceæ*), bearing their natural glandular trichomes.

Constituents.—Volatile oil; resin; bitter acid, humulotannic acid; phlobaphene, lupuline (an alkaloid). The volatile oil consists of a

sesquiterpene, *humulene*, 66 per cent., some olefinic terpenes, oxy-generated products, etc.

Remarks.—Hops are strobiles of *Humulus Lupulus*, the strobile being the chaffy, scale-like form in which the flowers of hops arrange themselves. They are somewhat similar to the pine-cone, and at the base of each scale the fruit is found—in the form of a minute nutlet. The inner surface of the chaffy scales of hops are found covered with hair-like appendages called by the pharmacopœia “glandular trichomes,” and these trichomes, shaken out from the dry hops, constitute lupulin, which is official. The medical action of these two drugs is, therefore, quite similar. Hops are chiefly used in the manufacture of beer.

Medical Properties.—Tonic and hypnotic, chiefly in domestic practice,—hop poultice, etc.,—as in medicine the more active lupulin is preferred.

Dose.—2 Gm. (30 grains).

LUPULINUM. Lupulin.

The glandular trichomes separated from the fruit of *Humulus Lupulus* Linné (Fam. *Moraceæ*).

Constituents.—Identical with those of hops and in more concentrated form.

Medical Properties.—Like hops. Administered as *oleoresin*, U.S. P. (p. 262), or as *fluidextract*, U. S. P.

Dose.—500 milligrammes (7½ grains).

DRUGS CONTAINING VOLATILE OIL, RESIN, AND BITTER PRINCIPLE

Drugs of this class closely resemble those just considered, the only difference being that while in the class just considered the aromatic feature prevailed, the drugs now to be taken are bitter rather than aromatic. The bitter principles found in these drugs have not in most cases been carefully studied.

PYRETHRUM. Pyrethrum. Pellitory.

The root of *Anacyclus Pyrethrum* (Linné) De Candolle (Fam. *Compositæ*).

Constituents.—Acrid resin; fixed oil; much inulin; no starch; pyrethrine (an alkaloid).

Remarks.—Pellitory scarcely belongs in this class of drugs, as there is no mention in literature as to its containing volatile oil, nor is it strikingly bitter.

Medical Properties.—Sialagogue and irritant. It is seldom used, but is said to be efficient in toothache. The *tincture* (p. 237) is official.

Dose.—Of drug, 2 Gm. (30 grains).

CYPRIPEDIUM. Cypripedium.

The dried rhizome and roots of *Cypripedium hirsutum* Miller (*Cypripedium pubescens* Willdenow) or of *Cypripedium parviflorum* Salisbury (Fam. *Orchidaceæ*).

Constituents.—Volatile oil, resins, volatile acid, bitter glucoside, etc.

Medical Properties.—Antispasmodic. Administered as *fluidextract*, U. S. P., p. 247.

Dose.—1 Gm. (15 grains).

SALVIA. Salvia. Sage.

The dried leaves of *Salvia officinalis* Linné (Fam. *Labiatae*).

Constituents.—Volatile oil, tannin, etc.

The *volatile oil* consists of thujone, 50 per cent.; pinene, cineol, borneol, etc.

Medical Properties.—Carminative, astringent, flavor. No official preparations.

Dose.—2 Gm. (30 grains).

EUPATORIUM. Eupatorium.

The dried leaves and flowering tops of *Eupatorium perfoliatum* Linné (Fam. *Compositae*).

Constituents.—Volatile oil, bitter principle, *eupatorin*, etc.

Medical Properties.—Bitter, stomachic. The *fluidextract* (p. 247) is official.

Dose.—2 Gm. (30 grains).

MARRUBIUM. Marrubium. Horehound.

The dried leaves and flowering tops of *Marrubium vulgare* Linné (Fam. *Labiatae*).

Constituents.—Volatile oil, resin, *marrubiin* (a bitter principle melting at 160° C.), which is the lactone of *marrubic acid*, $C_{20}H_{28} \begin{smallmatrix} \text{COOH} \\ \text{OH} \end{smallmatrix}$.

Medical Properties.—Stimulant, tonic, popular domestic cough remedy.

Dose.—2 Gm. (30 grains).

ANTHEMIS. Anthemis. Roman Chamomile. English Chamomile.

The dried flower-heads of *Anthemis nobilis* Linné (Fam. *Compositae*), collected from cultivated plants.

Constituents.—Volatile oil; anthemic acid; a glucoside. The volatile oil consists of a mixture of the esters of isobutyl, amyl, and hexyl alcohols with butyric and angelic acids.

Medical Properties.—Carminative, stimulant, emmenagogue.

Dose.—2 Gm. (30 grains).

MATRICARIA. Matricaria. German Chamomile.

The dried flower-heads of *Matricaria Chamomilla* Linné (Fam. *Compositae*).

Constituents.—Volatile oil, which is blue in color; *anthemic acid*.

Medical Properties.—Like anthemis.

Dose.—16 Gm. (240 grains).

CALENDULA. Calendula. Marigold.

The dried ligulate florets of *Calendula officinalis* Linné (Fam. *Compositæ*).

Constituents.—Volatile oil; resin; bitter principle; calendulin (inert).

Remarks.—*Calendula* belongs to the largest of all plant families, the *Compositæ*, or sunflower family. The type of the family is the sunflower, the large so-called “flower” of which is really a large collection of small flowers arranged in a close cluster or head. An examination of the sunflower will show this: the black, brush-like disc being composed of florets of one sort (the tubular), while the outer row is composed of florets with a large yellow expansion of the petal on one side. As this expansion is somewhat strap-shaped, such little flowers as have them are called ligulate, from the Latin *ligula*, a strap. Flower-heads, like the sunflower, being compounded of many little flowers, are grouped into a plant family called the *Compositæ*. To this family belong the two chamomiles and arnica, and an examination of any of these will show the many little florets that go to make up the flower-head, while the official *calendula* consists of the ligulate florets only.

ARNICA. Arnica.

[ARNICÆ FLORES, PHARM. 1890]

The dried flower-heads of *Arnica montana* Linné (Fam. *Compositæ*).

Constituents.—Volatile oil; resin; *arnicin*; *arnicine*.

Medical Properties.—Irritant and vulnerary. Used chiefly externally in the form of the official *tincture* (p. 222).

Dose—Of drug, 1 Gm. (15 grains).

Arnica root, U. S. P. 1890, contain about the same constituents as the flowers and is used for the same purpose.

OLEORESINS AND BALSAMS

In this class we group those drugs which are plant exudations and consist of a mixture of volatile oil and resin (*natural oleoresins*) or of an oleoresin containing benzoic, cinnamic, and other aromatic acids (*balsams*). We have already studied (p. 259) pharmaceutical oleoresins, those bodies prepared by extracting the oil and resins from such plant drugs as we have just considered—*e. g.*, capsicum, cubeb, etc.—by percolation with acetone, ether, or alcohol. The oleoresins we will now consider are natural exudations, oozing from trunks of trees through natural fissures or artificial incisions.

TEREBINTHINA. Turpentine.

A concrete oleoresin obtained from *Pinus palustris* Miller, and from other species of *Pinus* (Fam. *Pinaceæ*).

Constituents.—Volatile oil, 25 per cent.; resin, 70 per cent. (rosin, U. S. P.).

Remarks.—This substance is familiarly and incorrectly called “gum turpentine.” Already it has been mentioned that the modern

idea of a gum is a substance soluble in water and insoluble in alcohol. As turpentine is insoluble in water and soluble in alcohol, it is clear it should no longer be considered among the "gums."

We will consider rosin in the chapter devoted to Resinous Bodies, at which time its chemistry will be discussed.

Medical Properties.—Stimulant. Used almost entirely externally as plaster base.

OLEUM TEREBINTHINÆ. Oil of Turpentine.

A volatile oil recently distilled from turpentine. It should be kept in well-stoppered bottles.

A thin, colorless liquid, having a characteristic odor and taste, both of which become stronger and less pleasant by age and exposure to the air.

Specific gravity: 0.860 to 0.870 at 25° C. (77° F.).

When Oil of Turpentine is distilled, the larger part should pass over between 155° and 162° C. (311° and 323.6° F.).

Soluble in 3 times its volume of alcohol.

If 5 Cc. of Oil of Turpentine be shaken with an equal volume of potassium hydroxide T.S., its color should not become darker than a light straw-yellow upon standing twenty-four hours.

If 1 Cc. of the Oil be evaporated in a small dish on a water-bath, it should leave not more than a very slight residue (absence of *petroleum*, *paraffin oils*, or *rosin*).

Three drops of Oil of Turpentine, placed on a sheet of clean white filter-paper and exposed to the air, should evaporate entirely without leaving a permanent stain (absence of *kerosene* or *rosin oil*).

If 5 Cc. of Oil of Turpentine be placed in a small beaker, and 20 Cc. of sulphuric acid be gradually added, with agitation, while the beaker is cooled by immersion in cold water, and the contents, after cooling and renewed agitation, be transferred to a burette, graduated in tenths, the clear layer which forms after the dark mass has settled should not measure more than 0.35 Cc. (absence of *petroleum benzin*, *kerosene*, or *similar hydrocarbons*).

Composition.—Chiefly pinene. The American oil is dextrogyrate, while the French oil is lævogyrate. Otherwise the two are practically identical.

Medical Properties.—Stimulant, antiseptic, urinary disinfectant. The pharmacopœia directs that for internal use the *rectified oil* should be employed.

OLEUM TEREBINTHINÆ RECTIFICATUM. Rectified Oil of Turpentine.

Recipe.—Oil of Turpentine, a convenient quantity.

Solution of Sodium Hydroxide, a sufficient quantity.

Shake the Oil of Turpentine thoroughly with an equal volume of Solution of Sodium Hydroxide, and introduce the mixture into a copper still connected with a well-cooled condenser. Recover about *three-fourths* of the Oil by distillation, separate the clear Oil from the water, and filter. Keep the product in well-stoppered, amber-colored bottles, in a cool place.

Rectified Oil of Turpentine should always be dispensed when oil of turpentine is required for internal use.

A thin, colorless liquid, which should conform to the properties and tests given under *Oleum Terebinthinæ*.

Specific gravity: 0.860 to 0.865 at 25° C. (77° F.).

If about 10 Cc. of the Oil be evaporated in a dish on a water-bath, no weighable residue should be left.

Remarks.—It will be seen that this represents the commercial oil mixed with alkali and redistilled, and it is thus freed from acids (formic, etc.) which are present in the crude oil, giving it irritating properties.

Medical Properties.—That of the crude oil.

Dose.—1 Cc. (15 minims).

The *emulsion* (p. 271) is official.

TEREBINTHINA CANADENSIS. Canada Turpentine. Balsam of Fir.

A liquid oleoresin obtained from *Abies balsamea* (Linné) Miller (Fam. *Coniferae*).

Composition.—Volatile oil, resin, bitter principle.

The *volatile oil* is chiefly *laevogyrate pinene*.

Medical Properties.—External stimulant. A constituent of *flexible collodion*, U. S. P. (p. 209).

Used largely for mounting microscopic specimens, the object being embedded in a hard, transparent varnish after standing.

Venice Turpentine.—Was originally the oleoresin from *Larix decidua*, the larch, a tree closely related to the pine. The product sold in American drug-stores for "drawing out" splinters saw neither Venice nor the larch tree, but is made by dissolving rosin in oil of turpentine.

PIX LIQUIDA. Tar.

A product obtained by the destructive distillation of the wood of *Pinus palustris* Miller, or of other species of *Pinus* (Fam. *Pinaceae*).

Constituents.—Volatile oil, U. S. P.; pitch; pyroligneous acid (p. 645).

Remarks.—Tar is made by stacking billets of pine wood on the earth into a conical mound, and setting fire to the wood after covering with plastered earth. Enough air is admitted through holes in the earth covering to permit the wood to smoulder, and a hole at the top of the mound serves as a chimney. Before stacking the wood, radiating ditches are dug on the ground on which the mound is to be built, the ditches running into a circular trench surrounding the mound. The smouldering heat decomposes the wood into smoke, which passes off through the hole in the top of the mound; tar, which runs into the circular trench and is dipped up into barrels; and charcoal, which is sacked after the fire is extinguished and the earth covering removed. The process has already been touched upon on pp. 83 and 406.

Medical Properties.—Stimulant and antiseptic. Used for bronchial affections as the official *syrup* (p. 197), and externally as the official *ointment* (p. 348).

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Glycerole of tar (N. F.) is made by mixing washed tar with alcohol, triturating mixture with magnesium carbonate and water, and filtering. It contains the equivalent of 63 Gm. tar to the liter. A simpler recipe consists in mixing 1 fluidounce oil of tar with 2 fluidounces of alcohol, triturating to smooth paste with 6 drachms magnesium carbonate, adding 4 fluidounces glycerin and 4 fluidounces water, macerating several days, and then filtering. (See p. 197.)

(2)

OLEUM PICIS LIQUIDÆ. Oil of Tar.

A volatile oil distilled from tar.

An almost colorless liquid when freshly distilled, but soon acquiring a dark reddish-brown color, and having a strong, tarry odor and taste.

Specific gravity: about 0.965 at 25° C. (77° F.).

It is readily soluble in alcohol, the solution showing an acid reaction to litmus-paper.

Composition.—A vast number of products similar to those obtained in the manufacture of crude pyroligneous acid, such as toluene, xylene, mesitylene, phenol, creosote, naphthalene, etc.

Medical Properties.—Stimulant and antiseptic.

Dose.—0.2 Cc. (3 minims).

OLEUM CADINUM. Oil of Cade.

A product of the dry distillation of the wood of *Juniperus Oxycedrus* Linné (Fam. *Coniferæ*).

A brownish or dark brown, clear, thick liquid, having a tarry odor, and an empyreumatic, burning, somewhat bitter taste.

It is almost insoluble in water, but imparts to it an acid reaction; it is only partially soluble in alcohol, but it is completely soluble in ether.

Composition.—Phenols, resin, large amount of cadinene.

Medical Properties.—Like tar, chiefly externally.

COPAIBA. Copaiba. Balsam of Copaiva.

An oleoresin derived from one or more South American species of *Copaiba* (Fam. *Leguminosæ*).

A pale yellow to brownish-yellow, more or less transparent and viscid liquid, sometimes fluorescent; having a peculiar, aromatic odor and a persistent, bitter, and acid taste.

Specific gravity: 0.950 to 0.995 at 25° C. (77° F.).

Insoluble in water; soluble or showing at most a slight opalescence in absolute alcohol, carbon disulphide, petroleum benzin, and in fixed and volatile oils; completely soluble in chloroform and ether.

When heated on a water-bath, it should evolve no odor of turpentine, and after forty-eight hours should leave a resinous mass weighing not less than 50 per cent. of its original weight.

One Gm. of Copaiba, when dissolved in 50 Cc. of alcohol, should require not less than 2.3 Cc., and not more than 2.8 Cc. of half-normal alcoholic potassium hydroxide for neutralization, using 1 Cc. of phenolphthalein T.S. as indicator (presence of a normal proportion of *acid resin*).

When 4 drops of nitric acid (sp. gr. 1.40) and 1 Cc. of glacial acetic acid are mixed in a test-tube, and 4 drops of Copaiba are carefully poured on top of the liquid, no reddish zone should appear; nor should the fluid assume a red or purple color after being shaken (absence of *gurjun balsam*).

If 5 Cc. of Copaiba be shaken with 15 Cc. of alcohol, and heated to boiling for one minute, no drops of oil should separate after cooling and standing for an hour (absence of *paraffin oils*).

If 20 drops of Copaiba be boiled with 1 Cc. of an alcoholic potassium hydroxide solution (1 in 10) for two minutes and cooled, and then twice its volume of ether be added to the liquid, no gelatinization should occur (absence of *fixed oils*).

If 1 Gm. of Copaiba be shaken with 10 Cc. of ammonia water in a stoppered vial, and allowed to stand for twenty-four hours, the liquid will become turbid, but it should not gelatinize, nor should a firm mass be formed (limit of *resin*).

Tests for Identity.—Given above.

Impurities.—Gurjun balsam, paraffin oils, fixed oils, excess of resin. Details above.

Constituents.—Volatile oil (80 per cent. in best quality), resin (p. 854).

Remarks.—Copaiba has been largely adulterated, hence lengthy tests given above. For making mass of copaiba, U. S. P. 1890, samples containing large amounts of resin should be employed, such as the Maracaibo copaiba, which contains only 30 per cent. to 40 per cent. oil.

Medical Properties.—Urinary antiseptic. Popular remedy for gonorrhea, administered in emulsion, such as copaiba mixture N. F.

Dose.—1 Cc. (15 minims).

OLEUM COPAIBÆ. Oil of Copaiba.

A volatile oil distilled from Copaiba. It should be kept in well-stoppered, amber-colored bottles, in a cool place, protected from light.

A colorless or pale yellow liquid, having the characteristic odor of copaiba, and an aromatic, slightly bitter, and pungent taste.

Specific gravity: 0.895 to 0.905 at 25° C. (77° F.), increasing with age. It is levogyrate.

Soluble in 2 volumes of alcohol.

Composition.—Caryophyllene; other constituents uncertain.

Medical Properties.—Similar to copaiba, but the oleoresin is considered more useful.

Dose.—0.5 Cc. (8 minims).

OFFICIAL BALSAMS

Balsams are natural products, differing from oleoresins only in possessing benzoic and cinnamic acids, the presence of which give the balsams their delightful odor. As all so-called "gums" are not true gums, even so every substance termed a balsam does not come within the pharmacæutic meaning of that term. For instance, copaiba is not a true balsam, since it contains neither benzoic nor cinnamic acid. It is an oleoresin pure and simple.

BALSAMUM PERUVIANUM. Balsam of Peru.

A balsam obtained from *Toluifera Pereiræ* (Royle) Baillon (Fam. *Leguminosæ*).

A viscid liquid of a dark brown color, free from stringiness or stickiness; transparent and reddish-brown in thin layers; of an agreeable vanilla-like odor and a bitter acrid taste, with a persistent after-taste. When swallowed it leaves a burning sensation in the throat. It does not harden on exposure to the air.

Specific gravity: 1.140 to 1.150 at 25° C. (77° F.).

Completely soluble in absolute alcohol, chloroform, and glacial acetic acid; only partially soluble in ether and petroleum benzin; soluble in 5 parts of alcohol, with not more than a slight opalescence. Water, when agitated with the Balsam, shows an acid reaction to blue litmus-paper.

If 10 drops of the Balsam be triturated with 20 drops of sulphuric acid, a tough, homogeneous, brownish-red mass should result, which, when washed with cold water, should develop a violet color upon its surface, and, when drained, be converted into a brittle resinous mass (absence of *fixed oils*).

If 1 Gm. of the Balsam be shaken with 5 Cc. of petroleum benzin, the mixture warmed on a water-bath for ten minutes, and a sufficient quantity of the solvent be added to replace loss by evaporation, then if 2 Cc. of the benzin solution be evaporated and treated with a drop of nitric acid (sp. gr. 1.42), a permanent green or bluish-green color should not be produced (absence of *rosin*).

The remaining 3 Cc. of the benzin solution, when shaken with an equal volume of an aqueous solution of copper acetate (1 in 1000), should not be colored green or bluish-green (absence of *rosin*, *turpentine*, *storax*, *fatty oils*, etc.).

On mixing the Balsam with half its volume of calcium hydroxide and heating for half an hour on a water-bath, a solid mass should not be formed (absence of *rosin*, *storax*, or *copaiba*).

If 1 Gm. of the Balsam be dissolved in 100 Cc. of alcohol and 1 Cc. of phenolphthalein T.S. be added, not more than 2 Cc. of half-normal alcoholic potassium hydroxide should be required to produce a pink color (limit of *acid resins*).

Mix 3 Gm. of the Balsam with 30 Cc. of sodium hydroxide T.S. and shake the mixture for a few minutes with 60 Gm. of ether. Transfer 51.5 Gm. of the ether-solution to a flask and evaporate to dryness. The residue, when dried to constant weight by a gentle heat, should weigh not less than 1.4 Gm. (presence of at least 56 per cent. of *cinnamein*). If this residue be dissolved in 25 Cc. of alcohol, then mixed with 25 Cc. of half-normal alcoholic potassium hydroxide V.S., and heated carefully during half an hour on a water-bath, it should require not more than 13.2 Cc. of half-normal hydrochloric acid V.S. to exactly neutralize the liquid, 1 Cc. of phenolphthalein T.S. being used as indicator.

Tests for Identity.—Given above.

Impurities.—Fixed oils, rosin, turpentine, storax, fatty oils, copaiba, excess of acid resins. Details above.

Assay.—Cinnamein, 56 per cent. Details above.

Constituents.—Volatile oil (called *cinnamein*), 60 per cent.; resin, 30 per cent.; benzoic and cinnamic acids; vanillin, etc.

The *volatile oil* consists of benzyl alcohol, benzylic benzoate, and benzylic cinnamate.

The *resin* yields 66 per cent. protocatechuic acid, on fusing with potassium hydroxide.

Remarks.—Of the tests given above, reference need be made only to the cinnamein assay, which concluded with estimation of its saponification number, performed like the assay of oil of peppermint (p. 1043).

Medical Properties.—Stimulant, antiseptic. Chiefly externally.

Dose.—1 Gm. (15 grains).

BALSAMUM TOLUTANUM. Balsam of Tolu.

A balsam obtained from *Toluijera Balsamum* Linné (Fam. *Leguminosæ*).

A yellowish-brown, plastic solid, becoming brittle when old, dried, or exposed to cold. It is transparent in thin layers, has a pleasant, aromatic odor, recalling that of vanilla, and a mild, aromatic taste.

Readily soluble in alcohol, the solution showing an acid reaction to blue litmus-paper; also soluble in chloroform and the solutions of the fixed alkalies; almost completely soluble in ether, but nearly insoluble in water and petroleum benzin; partially soluble in carbon disulphide.

If 0.5 Gm. of the Balsam be shaken with 25 Cc. of carbon disulphide and allowed to stand for thirty minutes, and the liquid then filtered, the residue obtained by evaporating the filtrate to dryness, when dissolved in glacial acetic acid, should not yield a green color on the addition of a few drops of sulphuric acid (absence of *rosin*).

If 1 Gm. of the Balsam be shaken with 8 Cc. of petroleum benzin for five minutes, the supernatant liquid should not be colored green when shaken with an equal volume of an aqueous solution of copper acetate (1 in 1000) (absence of *rosin* and *copaiba*).

If to 1 Gm. of the Balsam dissolved in 50 Cc. of alcohol, 1 Cc. of phenolphthalein T.S. be added, not less than 4 Cc. nor more than 6 Cc. of half-normal alcoholic potassium hydroxide V.S. should be required to produce a red color (limit of *acidity*); if to this liquid more half-normal alcoholic potassium hydroxide V.S. be added, until the total amount has reached exactly 20 Cc., and the liquid heated in a water-bath for half an hour, and allowed to cool, then not less than 13.2 Cc. nor more than 14.5 Cc. of half-normal sulphuric acid V.S. should be required to neutralize the excess of potassium hydroxide V.S., phenolphthalein T.S. being used as indicator (limit of *saponifiable substances*).

Tests for Identity.—Given above.

Impurities.—Rosin, copaiba, excess of acidity and of saponifiable substances. Details above.

Constituents.—Volatile oil; two resins; benzoic and cinnamic acids; esters of these two acids with benzyl alcohol. The composition of volatile oil not known.

Medical Properties.—Flavor, stimulant, popularly used as an expectorant in the form of the official *syrup* (p. 202), which is made from the official *tincture* (p. 237). A constituent of *compound tincture of benzoin* (p. 223).

Dose.—1 Gm. (15 grains).

STYRAX. Storax.

A balsam obtained from the wood and inner bark of *Liquidambar orientalis* Miller (Fam. *Hamamelidaceæ*).

A semisolid, grayish, sticky, opaque mass, depositing, on standing, a heavy, dark brown stratum; transparent in thin layers and having an agreeable odor and a balsamic taste.

Insoluble in water, but completely soluble (with the exception of accidental impurities) in an equal weight of warm alcohol.

If the alcoholic solution, which has an acid reaction, be cooled, filtered, and evaporated, it should leave not less than 70 per cent. of the original weight of the balsam, in the form of a brown, semiliquid residue almost completely soluble in ether and in carbon disulphide, but insoluble in petroleum benzin.

When heated on a water-bath, Storax becomes more fluid, and if it be then agitated with warm petroleum benzin, the supernatant liquid, on being decanted and allowed to cool, will be colorless, and will deposit white crystals of cinnamic acid and cinnamic esters.

Constituents.—Volatile oil; resin; benzoic and cinnamic acids; *styracine* (cinnamate of cinnamyl) and other ethers; vanillin; *cinnamene* (or *styrol*), $C_6H_5CH=CH_2$.

The *volatile oil* consists of cinnamene, styrocamphene, and esters of cinnamic acid.

Remarks.—This product, while quite similar to the “sweet gum” of the southern States, is not identical therewith, the latter being the exudation of *liquidambar styraciflua*.

Medical Properties.—Similar to balsam of Peru. A constituent of *compound tincture of benzoin* (p. 237).

Dose.—1 Gm. (15 grains).

CHAPTER XLVIII

RESIN ACIDS AND NEUTRAL PRINCIPLES

THIS chapter deals with plant principles, of which less is known than any other group of chemical substances—in truth, the only reason why these bodies are grouped into this chapter is because they have not been sufficiently studied to know to what classes of chemicals they belong, and as its composition is worked out, each body will be

placed in its appropriate position in systematic chemistry, leaving nothing for discussion in this chapter.

This work is already begun. It is now known that resin of jalap owes its activity to the glucoside, convolvulin; while resin of scammony is active because of its glucoside, scammonin; therefore, these two so-called resins are considered among the glucosidal drugs.

The phrase "resin acid" is here used in the attempt to classify the important constituent found in the several plant products which we call *resins*. These bodies are best defined as plant substances soluble in alcohol, insoluble in water, and dissolving in alkalis to form "resin soaps." Hence they are acid in character. Whether all the resins considered in this chapter will on investigation comply with this definition remains to be seen, and let it be distinctly understood that practically all resins are far more complex than the above definition implies.

Tschirch has attempted a classification of resins as follows:

Resin Esters or Their Saponification Products: (Resines.)

These esters are combinations of aromatic acids, such as benzoic, cinnamic, and ferulic acids, with resin alcohols (resinols).

The resin alcohols can be grouped into two classes, according as they do or do not give the tannin reaction, the latter class being called resin-tannols.

Among the tannin-free resinols may be mentioned *storesin* in storax; while as examples of resin tannols may be cited *toluresin tannol* in balsam of tolu.

Resinol Acids:

Resin acids, such as abietinic acid in rosin, guaiaconic acid in guaiac, and copaivic acid in copaiba.

Resenes:

Indifferent substances of unknown composition, such as *dracoresene* in dragon's blood.

Resins are sometimes extracted from drugs by means of solvents (example: the resin from phytolacca); sometimes represent practically entire plant exudation (example: mastic); frequently exude from plants combined with volatile oils, then forming the class of drugs called *oleoresins* (example: turpentine, U. S. P.); and often exuding mixed with gummy substances, such an exudation being called a *gum-resin* (example: asafetida). Occasionally, the resin which exudes is aromatic by reason of the benzoic and cinnamic acid it contains, the example of such a *balsamic resin* being benzoin (p. 855), while natural oleoresin similarly flavored with benzoic and cinnamic acid is called a *balsam* (example: balsam of tolu (p. 850)). The last two classes of drugs as well as the oleoresins were discussed in the preceding chapter. It is advisable right here again to call attention to the distinction between natural and pharmaceutic oleoresins, already discussed on p. 259.

Neutral principles are plant principles of whose structural chemistry—of whose position in systematic chemistry—we know little or nothing. They are usually bitter and crystalline, are neither basic like alkaloids nor derivatives of glucose, as are the glucosides. That the grouping is purely artificial and temporary is shown by the fact that the most carefully studied neutral principle, santonin, is now known to be a lactone derived from hexahydrodimethyl-naphthalin

and will soon be considered in text-books among the naphthalin derivatives, even as coumarin, a lactone of oxycinnamic acid, is studied among the derivatives of allyl-benzene (p. 797).

It is apparent, after these preliminary remarks, that it is useless to discuss the chemistry of resins and of neutral principles; so we will proceed forthwith to a discussion of the drugs containing these bodies.

RESINS, GUM-RESINS, AND DRUGS CONTAINING NEUTRAL PRINCIPLES

OFFICIAL RESINS

GUAIAIACUM. Guaiac.

The resin of the wood of *Guaiacum officinale* Linné, or of *Guaiacum sanctum* Linné (Fam. *Zygophyllaceæ*).

Usually in irregular masses; externally greenish-gray brown; brittle, the fracture having a glassy lustre, in recent Guaiac yellowish-green or reddish-brown; transparent in thin splinters; fusible; odor balsamic; taste slightly acrid.

The powder is grayish, turning green on exposure to air.

Not more than 15 per cent. of Guaiac is insoluble in alcohol, and the alcoholic solution becomes blue on the addition of tincture of ferric chloride; acid number not less than 70 nor more than 80; ash not more than 4 per cent. The filtrate obtained on macerating the powder with 4 or 5 times its weight of petroleum benzin should be colorless, and should not give a green color on the addition of an equal volume of solution (1 in 1000) of cupric acetate (absence of *rosin*).

Tests for Identity.—Given above.

Acid Number.—70 to 80. Details in Part V.

Impurity.—Rosin. Details above.

Constituents.—*Guaiaconic acid*, $C_{19}H_{20}O_5$; *guaiaretic acid*, $C_{20}H_{26}O_4$, beta-resin, *guaiacic acid*.

These acids are typical "resin acids" (p. 852).

Remarks.—The alcoholic solution is characterized by turning blue in the presence of oxidizing agents, such as ferric chloride (U. S. P. test).

By reason of this property filter-paper steeped in tincture of guaiac is used as a test-paper for ozone and for blood, the hematin of the latter acting as the oxidizing agent.

Medical Properties.—Alterative in syphilis and rheumatism. The *tincture* (p. 228) and the *ammoniated tincture* (p. 228) are official.

Dose.—Of drug, 1 Gm. (15 grains).

Guaiac wood, the wood from which the above resin is derived, was recognized in the pharmacopœia of 1890, but is no longer official. It owes its properties almost entirely to the resin, although it is said to also contain a saponin.

MASTICHE. Mastic.

A concrete resinous exudation from *Pistacia Lentiscus* Linné (Fam. *Anacardiaceæ*).

In subglobular, lenticular, elongated or pear-shaped tears, about 3 Mm. in diameter, pale yellow or greenish-yellow, transparent, having a glass-like lustre, the surface sometimes very slightly dusty; brittle, becoming plastic when chewed; odor slight, balsamic; taste mild, terebinthinate.

Mastic is completely soluble in ether and almost completely soluble in alcohol.

Tests for Identity.—Given above.

Acid Number.—Not less than 65. Details in Part V.

Constituents.—*Mastichin*, $C_{20}H_{31}O$ (or beta-resin); *mastichic acid*, $C_{20}H_{29}O_2$ (or alpha-resin); volatile oil, 2 per cent.

Remarks.—The pharmacopœia requires an *acid number* of not less than 65 for this drug.

An explanation of this standard for resins is given under Volumetric Analysis (p. 1045).

Medical Properties.—Carminative. Ingredient of *pills of aloes and mastic* (p. 322).

Dose.—Of drug, 2 Gm. (30 grains).

Burgundy pitch is no longer official, having been dropped in the last revision. It is a resin prepared from a species of fir, and is used in medicine exclusively for the preparation of plasters.

Resin of copaiba (U. S. P. 1890), while considered as a pharmaceutical on p. 293, is worthy of a few lines devoted to its chemistry. It is the residue left on distillation of official copaiba, and, like all true resins, consists largely of a solid acid—in this case, *copaivic acid*. As mentioned on p. 312, this acid combines with moist magnesium oxide to form a salt, and to this action is due the solidification of oleoresin in making mass of copaiba.

RESINA. Rosin.

The residue left after distilling off the volatile oil from turpentine.

Usually in sharp, angular fragments, translucent, amber-colored, usually covered with a yellowish dust; at ordinary temperatures brittle, pulverizable; fracture shiny and shallow-conchoidal; odor and taste faintly terebinthinate.

The specific gravity of Rosin is 1.070 to 1.080; it is easily fusible, and burns with a dense yellowish smoke, yielding no appreciable ash; soluble in alcohol, ether, benzene, carbon disulphide, acetic acid, fixed or volatile oils, and in solutions of potassium or sodium hydroxide; acid number not less than 150.

Tests for Identity.—Given above.

Acid Number.—Not less than 150. Details in Part V.

Constituents.—Chiefly *abietic acid anhydride*. Abietic acid, $C_{19}H_{29}O_2$, is obtained by digesting rosin with 70 per cent. alcohol, freeing the solution of impurities by filtration, and then dissolving the residue in 92 per cent. hot alcohol, from which the abietic acid crystals separate on cooling.

Remarks.—As already mentioned (p. 817), rosin is the resinous constituent of the natural oleoresin, *turpentine*, and is obtained by distilling the volatile oil—the so-called “spirit of turpentine”—therefrom.

Medical Properties.—Mild irritant. Use chiefly externally as constituent or plasters, cerates, and ointments. Official preparations: *Rosin cerate* (p. 342), *compound rosin cerate* (p. 342). An ingredient of adhesive plaster of the pharmacopœia of 1890.

Dose.—250 milligrammes (4 grains).

ELASTICA. Rubber.

The prepared milk-juice of several species of *Hevea Aublet* (Fam. *Euphorbiaceæ*), known in commerce as Para Rubber.

In flask-shaped or roundish masses, or in pieces of the same having sharply

incised surfaces and exhibiting a laminated structure; floating on water; externally brownish to brownish-black; internally of a lighter tint, mottled; odor creosote-like; nearly tasteless.

Pure Para Rubber is insoluble in water, dilute acids, or dilute solutions of the alkalies; soluble in chloroform, carbon disulphide, oil of turpentine, petroleum benzin, and benzene; when heated to about 125° C. (257° F.) it melts, remaining soft and adhesive after cooling.

Constituents.—Isoprene, C_5H_8 ; caoutchín (dipentene), $C_{10}H_{16}$.

Remarks.—The "preparation" of the milk juice mentioned in the official definition consists of submitting the fresh juice to the action of smoke made by burning fatty fuel,—like palm fruit,—which is allowed to smoulder with but slight access of air. The milk juice is held over the smoke on a wooden paddle, which has been dipped into a bucket containing the juice, and by deft manipulation the liquid juice is prevented from dripping from the paddle until it solidifies. The paddle containing the dried rubber is again dipped into the fresh juice, and the paddle once more rotated over the smoke until that layer has solidified, and this process continued until the paddle holds a large mass of rubber.

As mentioned in the official description, when heated to above 125° C., it becomes a sticky mass, and the products made from it should, therefore, be kept from undue heat.

Rubber is made more durable by the process of vulcanization. In making soft rubber it is kneaded with 10 per cent. sulphur (or a metallic sulphide) at a temperature of 120° to 136° C., while in making hard rubber the crude rubber is heated with 30 to 35 per cent. sulphur to 140° to 142° C., and further hardened with rosin, chalk, etc.

The official drug is the pure native unvulcanized variety, and that of the best commercial grade—Para rubber.

Medical Use.—Plaster base (p. 352). A constituent of *adhesive plaster*, U. S. P. (p. 355).

BENZOINUM. Benzoin.

A balsamic resin obtained from *Styrax Benzoin* Dryander, and another unidentified species of *Styrax* (Fam. *Styracæ*).

In pebble-like bodies or tears, mostly 0.5 to 5 Cm. long and about one-fourth as thick, slightly flattened, straight or curved, yellowish- to rusty-brown externally, milky-white on fresh fracture, separate or very slightly agglutinated (Siam Benzoin), or embedded in a dry resinous mass, which varies from reddish-brown to reddish-gray or grayish-brown; opaque or slightly translucent and more or less lustrous (Sumatra Benzoin); brittle, becoming soft on warming, and yielding benzoic acid on sublimation; odor agreeable, balsamic (vanilla-like in the Siam variety); taste slightly acid.

Benzoin is almost wholly soluble in 5 parts of warm alcohol, the solution showing an acid reaction to blue litmus-paper; soluble in solutions of sodium or potassium hydroxide. It should not, on incineration, yield more than 2 per cent. of ash.

Constituents.—Resin, benzoic acid, cinnamic acid, vanillin.

Remarks.—Benzoin would be considered a balsam, save that the quantity of volatile oil it contains is so small that its base can hardly be considered as an oleoresin. Accordingly, the pharmacopœia calls it "a balsamic resin."

Benzoin is interesting as the original source of benzoic acid, which thus received its name.

Note that the pharmacopœia permits the use of two commercial varieties of benzoin—Siam and Sumatra. Of these, the former possesses a delightful odor of vanilla, and is used by perfumers in preference to the less aromatic Sumatra variety. Medicinally, the two are about equal.

Medical Properties.—Expectorant. Used chiefly externally as an antiseptic and vulnerary. The *tincture* (p. 223) and the *compound tincture* (p. 223) are official. It is also used in making *benzoinated lard* (p. 733).

Dose.—1 Gm. (15 grains).

GUM-RESINS

Gum-resins are plant exudations consisting of gum and resin. We have already learned what a gum (p. 852) is, and wherein a resin (p. 704) differs from a gum, the pharmacæutic difference being one of solubility, resins dissolving in alcohols and being insoluble in water, while gums dissolve in water and not in alcohol. Again, resins are terpene derivatives, while gums are dissociation products of cellulose.

The gum of a gum-resin will not dissolve in alcohol; the resin will; and since the resin is the active constituent, tinctures of such drugs are made by maceration in 95 per cent. alcohol and filtration of the resinous solution from the undissolved gum. In water, the gum will dissolve and the resin will not, but by rubbing such a drug with water the mucilage formed by the dissolved gum emulsifies the resin,—also the volatile oil, if any be present,—making a milky mixture. Such a mixture is formed when *asafetida* is rubbed with water, and since the preparation is clearly a form of emulsion, it is so termed by the pharmacopœia. Up to the revision of 1890 they were grouped with the mixtures of the pharmacopœia. Three gum-resins are now official, *asafetida*, *gamboge*, and *myrrh*, and of these, the first is a representative of the *Umbelliferae*—that family of plants to which belong *anise*, *fennel*, *coriander*, and *caraway*.

ASAFETIDA. *Asafetida*.

A gum-resin obtained from the root of *Ferula fetida* (Bunge) Regel, and probably other species of *Ferula* (Fam. *Umbelliferae*).

In irregular masses composed of tears of variable size embedded in a yellowish-brown or reddish-brown matrix; when fresh, the tears are tough, yellowish-white and translucent, or milky-white and opaque, changing gradually on exposure to pinkish and finally reddish-brown; the freshly fractured surface becomes greenish on the application of a few drops of a 40 per cent. nitric acid solution; becoming hard and brittle by drying; odor persistent, alliaceous; taste bitter, alliaceous, and acrid.

When triturated with water, *Asafetida* yields a milk-white emulsion which becomes yellowish on the addition of ammonia water. Not less than 50 per cent. should dissolve in alcohol. When incinerated, *Asafetida* should yield not more than 10 per cent. of ash.

Constituents.—Resin, 65 per cent.; gum, 25 per cent.; bassorin; volatile oil.

The resin is chiefly *ferulaic acid* (p. 797), while the oil is a mixture of sulphides, $(C_6H_{11})_2S$ and $(C_6H_{11})_2S_2$, according to Hlasiwetz; or $C_7H_{14}S_2$, $C_{11}H_{20}S_2$, $C_8H_{16}S_2$, and $C_{10}H_{18}S_2$, with terpenes, according to Semmler.

Medical Properties.—Antispasmodic (in hysteria), carminative.

The *emulsion* (p. 269), *pills* (p. 323), and *tincture* (p. 222) are official, and the tincture is an ingredient of the popular colic cure, *Dewee's Carminative* (mixture of magnesia and asafetida, U. S. P. 1880). As this mixture contains opium, its use is not advisable, and some physicians specify the preparation "without opium," in which case it is made exactly as per U. S. P. 1880, save that the tincture of opium is omitted.

Dose.—Of drug, 250 milligrammes (4 grains).

CAMBOGIA. Gamboge.

A gum-resin obtained from *Garcinia Hanburii* Hooker filius (Fam. *Guttifera*).

Constituents.—Gum, 18 per cent.; resin, 80 per cent. (gambogic acid). The resin, on fusion with potassa, yields phloroglucin.

Medical Properties.—Hydragogue cathartic. Always in combination, since it is very drastic. An ingredient of *compound cathartic pills* (p. 323).

Dose.—125 milligrammes (2 grains).

MYRRHA. Myrrh.

A gum-resin obtained from *Commiphora Myrrha* (Nees) Engler (Fam. *Burseraceæ*).

Constituents.—Gum, 50 per cent.; resin, 35 per cent.; volatile oil, 2 to 4 per cent.

Medical Properties.—Carminative. The official *tincture* (p. 231) is a useful addition to mouth-washes.

Dose.—500 milligrammes (7½ grains).

Ammoniac, the gum-resin from *Dorema Ammoniacum*, was dropped in the last pharmacopœial revision. It is a stimulant, and given in doses of 1 Gm. (15 grains).

The following drugs are said to owe their activity to resinous matter not associated with volatile oil.

PHYTOLACCA. Phytolacca. Poke Root.

[PHYTOLACCÆ RADIX, PHARM. 1890]

The dried root of *Phytolacca decandra* Linné (Fam. *Phytolaccaceæ*), collected in autumn.

Constituents.—Resin, tannin, a glucoside, a volatile acid.

Medical Properties.—Alterative (in rheumatism); large doses, emetic. The *fluidextract* (p. 247) is official.

Dose.—As alterative, 125 milligrammes (2 grains). As emetic, 1 Gm. (15 grains).

Poke berries, the fruit of *Phytolacca decandra*, were official until the last revision, when the drug was dropped. It is emetic and cathartic, and of supposed value in reducing superfluous flesh. *Dose*.—1 Gm. (15 grains).

GOSSYPII CORTEX. Cotton Root Bark.

[GOSSYPII RADICIS CORTEX, PHARM. 1890]

The dried bark of the root of *Gossypium herbaceum* Linné, or of other cultivated species of *Gossypium* (Fam. *Malvaceæ*).

Constituents.—An acid resin, tannin, etc.

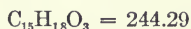
Remarks.—Cotton root bark is one of the official substances yielded by the cotton plant, the others being purified cotton and cotton seed oil. It is used as an emmenagogue, and should not be sold indiscriminately, being frequently used to produce abortion. This explains why, in the recent revision, the largely used *fluidextract of cotton root bark* was dropped.

Medical Properties.—Emmenagogue and ecbolic.

Dose.—2 Gm. (30 grains).

NEUTRAL PRINCIPLES AND DRUGS CONTAINING SAME

SANTONINUM. Santonin.



The inner anhydride or lactone of santonic acid, obtained from *Santonica*. It should be kept in dark amber-colored vials and in a dark place.

Colorless, shining, flattened rhombic prisms; odorless, and nearly tasteless when first put into the mouth, but afterward developing a bitter taste; permanent in the air, but turning yellow on exposure to light. It sublimes without decomposition and is soluble in alkalis and in most volatile and fatty oils. Santonin which has become yellow may be converted into white crystals by recrystallization from alcohol.

Soluble in 5300 parts of water, 34 parts of alcohol, 78 parts of ether, and in 2.5 parts of chloroform at 25° C. (77° F.); soluble in 800 parts of water at 80° C. (176° F.) and in 5 parts of alcohol at 60° C. (140° F.).

It melts at 170.3° C. (338.5° F.). When ignited, it is consumed, leaving no residue.

Its solutions are laevogyrate and neutral to litmus-paper.

If 0.5 Gm. of Santonin be heated with 5 Cc. of alcoholic potassium hydroxide T.S., a red color will be developed.

Upon shaking 0.01 Gm. of Santonin with a cold mixture of 1 Cc. each of sulphuric acid and water, heating to 100° C. (212° F.), and adding a minute trace of very dilute solution of ferric chloride, a violet color will result.

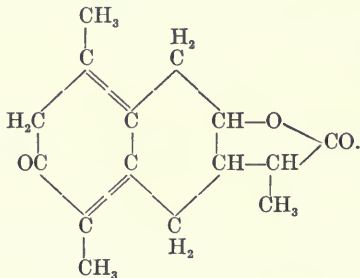
Sulphuric acid added to Santonin should not produce more than a faintly yellow color (absence of *sugar* and *other readily carbonizable organic impurities*).

If 2 Gm. of Santonin be boiled with 80 Cc. of water and 5 Cc. of diluted sulphuric acid, and the liquid, after frequent shaking, be allowed to become cold and then filtered, mercuric potassium iodide T.S., or iodine T.S., should produce no cloudiness in 10 Cc. of the filtrate, mixed with 10 Cc. of distilled water, even after standing for three hours (absence of *alkaloids*).

Tests for Identity. Given above.

Impurities.—Sugar, etc. Alkaloids. Details above.

Composition.—According to Cannizzaro, santonin is—



that is, a derivative of hexahydronaphthalene (p. 863).

Remarks.—The change to yellow on exposure to light is said to be due to its conversion into *photosantonin acid*, $C_{15}H_{22}O_5$. This explains the importance of keeping santonin and its preparations in a dark place.

Medical Properties.—Anthelmintic. Usually administered in the form of the official *troches* (p. 333).

Dose.—65 milligrammes (1 grain).

SANTONICA. *Santonica*. *Levant Wormseed*.

The dried unexpanded flower-heads of *Artemisia pauciflora* (Ledebour) Weber (Fam. *Compositæ*).

Constituents.—Santonin, 2 per cent.; volatile oil, 2 per cent. The volatile oil consists of *cineol* (p. 815) and *cajuputene* (or *cinene*).

Remarks.—*Santonica* is one of the two drugs called “wormseeds,” and neither are seeds. The other wormseed, *chenopodium*, is a fruit, while *santonica* is a tiny cluster of unopened flowers. The two are readily distinguished by rubbing the specimen between the thumb and forefinger. If *chenopodium*, a little chaff rubs off, leaving a minute black seed; if *santonica*, the entire specimen rubs into dry, chaffy scales. *Santonica* is important as the source of santonin.

Medical Properties.—Anthelmintic.

Dose.—2 Gm. (30 grains). No dose is given in the pharmacopœia, since the drug is now used almost exclusively as a source of santonin.

ELATERINUM. *Elaterin*.



A neutral principle obtained from *elaterium*, a substance deposited by the juice of the fruit of *Ecballium Elaterium* (Linné) A. Richard (Fam. *Cucurbitaceæ*).

Minute, white, hexagonal scales or prismatic crystals; without odor, and having a slightly acrid, bitter taste; permanent in the air, and containing no water of crystallization.

Insoluble in water; soluble in 262 parts of alcohol, 318 parts of ether, 22 parts of chloroform, 272 parts of benzene, and 200 parts of amyl alcohol at 25° C. (77° F.); soluble in 75 parts of alcohol at 60° C. (140° F.).

When heated to 190° C. (374° F.) it turns yellow, and at 216° C. (420.8° F.) it melts, forming a yellowish-brown liquid. On ignition, it is consumed without leaving any residue.

Its solutions are neutral to litmus-paper.

Sulphuric acid colors it yellow, the color changing gradually to scarlet.

Sulphuric acid containing a trace of ammonium vanadate produces a blue color, changing to green and then to brown.

Sulphuric acid containing a drop of formaldehyde gives a brown color.

Sulphuric acid containing a trace of potassium dichromate produces an olive-green color, gradually turning darker.

If a crystal of *Elaterin* be added to a little hydrochloric acid, and this evaporated to dryness, the residue washed with hot water, and afterward treated with sulphuric acid, a brownish-red (amaranth) color will be produced.

An alcoholic solution of *Elaterin* should not be precipitated by tannic acid T.S., mercuric chloride T.S., or platinic chloride T.S. (absence of, and difference from, *alkaloids*).

Tests for Identity.—Given above.

Color Tests.—Given above.

Impurity.—*Alkaloids*. Details above.

Composition.—While the pharmacopœia gives formula $C_{20}H_{28}O_5$, Thoms and Mann (1907), operating with a pure specimen, melting at $232^\circ C.$, found its formula, $C_{22}H_{30}O_6$. They find it has two lactone rings attached to a naphthalene nucleus.

Remarks.—Elaterin, according to the pharmacopœia, is “a neutral principle, obtained from elaterium.” This, in turn, is a constituent of the juice of the squirting cucumber, *Ecballium Elaterium*, which is a plant belonging to the gourd family. The pronunciation of the two words is different, the accent in the official name being on the second syllable (e-lat-er-in), while in the other word it is on the third syllable (el-a-ter-i-um).

Medical Properties.—Hydragogue cathartic. Usually administered in the form of the official *triturate* (p. 305).

Dose.—Of elaterin, 5 milligrammes ($\frac{1}{10}$ grain).

Picrotoxin, $C_{15}H_{16}O_6$, which was formerly official, is the active principle of *fish-berries* (*Anemirta paniculata*). It is a dangerous poison, and is used chiefly for killing body-vermin.

Dose.—1 milligramme ($\frac{1}{64}$ grain).

TARAXACUM. Taraxacum. Dandelion.

The dried root of *Taraxacum officinale* Weber (Syn. *Taraxacum Taraxacum* (Linné) Karsten) (Fam. *Compositæ*), collected in autumn.

Constituents.—*Taraxacin*; *taraxacerin*; inulin, etc.

Remarks.—Taraxacum is the root of that common weed, the dandelion. The latter synonym comes from the French “dent de lion,” the lion’s tooth, so termed after the jagged edges of its leaves. It is a good remedy in liver trouble. Look out for prescriptions containing fluidextract of taraxacum and nitrohydrochloric acid. Both are valuable hepatic stimulants, but when mixed they are apt to explode (p. 1079).

Note that the pharmacopœia directs that the drug be collected in the autumn. This is because the young root collected in the spring contains little or no bitter principle and much sugar. During the summer the latter is converted into the inulin found in the official drug.

Medical Properties.—Hepatic stimulant and digestive. Official preparations: the *extract* (p. 289) and the *fluidextract* (p. 250).

Dose.—8 Gm. (120 grains).

APOCYNUM. Apocynum. Canadian Hemp.

The dried rhizome of *Apocynum cannabinum* Linné, or of closely allied species of *Apocynum* (Fam. *Apocynaceæ*).

Constituents.—*Apocynin*; *apocynein*, *resin*, *tannin*, etc.

Apocynin is a resinous, bitter principle, while apocynein is a glucoside. Both act physiologically like digitalis.

Medical Properties.—Cardiac tonic and diuretic. Large doses, emetic. The *fluidextract* (p. 247) is official.

Dose.—1 Gm. (15 grains).

CALUMBA. Calumba. Columbo Root.

The dried root of *Jateorhiza palmata* (Lamarck) Miers (Fam. *Menispermaceæ*).

Constituents.—*Columbin*; *columbic acid*; three alkaloids, *columbamarin*, *jateorhizine*, and *palmitine*; starch; no tannin.

Columbin is a bitter principle, to which several formulas have been assigned by different investigators. Frey says it is $C_{27}H_{28}O_9$, and is a lactone. On treatment with alkali it yields columbic acid, $C_{28}H_{32}O_{10}$. The three alkaloids resemble berberine, and give the drug its yellow color.

Medical Properties.—Simple bitter. The *tincture* (p. 224) and *fluidextract* (p. 246) are official.

PODOPHYLLUM. Podophyllum. MandrakeRoot. May Apple Root.

The dried rhizome of *Podophyllum peltatum* Linné (Fam. *Berberidaceæ*).

Constituents.—*Picropodophyllin*; *picropodophyllinic acid*; *podophyllinic acid*; green oil; resin; starch, etc.

Picropodophyllin is a bitter principle, $C_{23}H_{24}O_9 \cdot 2H_2O$, while picropodophyllinic acid has the formula $C_{20}H_{24}O_9$, and is physiologically inactive. The two combine to form *podophyllotoxin*.

Podophyllum is the rhizome of an American plant commonly called *may apple*, or *mandrake*. It yields that important purgative, podophyllin, which is official under the name of *resin of podophyllum* (p. 292), since it is not a definite chemical, but a mixture of the active principles of the drug, cited above, associated with resin. Too great stress cannot be laid on the difference between *podophyllum*—the rhizome—and *podophyllin*—the trade name for resin.

Medical Properties.—Hydragogue cathartic. Usually administered with other purgatives. Official preparations. The *resin* (cited above) and the *fluidextract* (p. 246). The resin is a constituent of *vegetable cathartic pills*, U. S. P. (p. 323), and of *pills of podophyllum, belladonna, and capsicum*, U. S. P. (p. 325).

Dose.—Of drug, 500 milligrammes ($7\frac{1}{2}$ grains).

QUASSIA. Quassia.

The wood of *Picrasma excelsa* (Swartz) Planchon (Fam. *Simarubaceæ*), known commercially as Jamaica Quassia, or of *Quassia amara* Linné (Fam. *Simarubaceæ*), known commercially as Surinam Quassia.

Constituents.—Quassin, picrasmin, mucilage, resin, etc.

Remarks.—Quassia is the wood of the tree, *Picrasma excelsa*, and its bitterness is well known. Its use in the form of drinking-cups was quite popular some years since, but is no longer in vogue.

Medical Properties.—Bitter tonic. The *extract* (p. 288), the *tincture* (p. 234), and the *fluidextract* (p. 247) are official.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

SANTALUM RUBRUM. Red Saunders.

The heart-wood of *Pterocarpus santalinus* Linné filius (Fam. *Leguminosæ*).

Constituents.—*Santalin* (or *santalic acid*), a red coloring principle.

Medical Properties.—Used entirely as coloring agent, and employed for this purpose in *compound tincture of lavender* (p. 230).

EUONYMUS. Euonymus. Wahoo Bark.

The dried bark of the root of *Euonymus atropurpureus* Jacquin (Fam. *Celastraceæ*).

Constituents.—*Euonymin*, *euonic acid*, resin, starch, etc.

Medical Properties.—Laxative and diuretic. The *extract* (p. 283) and the *fluidextract* (p. 246) are official.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

CUSSO. Kousso.

[BRAYERA, U. S. P. 1880]

The dried panicles of the pistillate flowers of *Hagenia abyssinica* (Bruce) Gmelin (Fam. *Rosaceæ*).

Constituents.—Resinous principle, $C_{31}H_{38}O_{10}$, called *tænin*, or *kosin*; tannin, etc.

Remarks.—The pharmacopœia defines this drug as “the dried panicles of the pistillate flowers.” A panicle is a special form of flower cluster, while pistillate flowers are those containing the pistils or female organ and no stamens.

Each perfect flower has stamens, or male organs, which produce the pollen, and a pistil or pistils, which are fertilized by or absorb the pollen and which develop within their ovaries, the seeds. In some plants the male and female organs—the stamens and pistils respectively—are not found in the same flower, in which event the flowers containing the stamens are called the staminate flowers, while the flowers containing the pistils are called the pistillate flowers. The official drug, *cusso*, consists of the latter form of flower cluster.

Medical Properties.—Tenifuge. Usually administered in the form of an unstrained infusion, it being claimed that the action is largely mechanical.

Dose.—16 Gm. (240 grains).

LACTUCARIUM. Lactucarium.

The concrete milk-juice of *Lactuca virosa* Linné (Fam. *Compositæ*).

Constituents.—*Lactucin*, *lactucerin*, *lactucic acid*, *lactucopicrin*, acetates of *lactuceryl*, gum, resin, caoutchouc, or waxy substance.

Remarks.—This drug is the dried milk juice of *Lactuca virosa*, which is the wild lettuce, a plant closely allied to our garden lettuce, which, when plucked, exudes a milk juice. Such juice exuding from the wild lettuce, on drying, yields the official drug, which is a very useful sedative. Each lettuce plant naturally yields a comparatively small amount of juice, and when it is remembered that the dried juice must be scraped from each cut plant, the high price of *lactucarium* is explained.

The rubbery or waxy constituent of *lactucarium* can be extracted with benzine, which solvent does not remove the active constituents. Hence the use of benzin in preparing the official *tincture* (p. 229).

Medical Properties.—Mildly narcotic. Useful in hacking coughs. It is said that the proprietary *lactucarium* preparations contain morphine. From the official *tincture* (see above) the official *syrup* (p. 197) is prepared.

Dose.—Of drug, 1 Gm. (15 grains).

CHAPTER XLIX

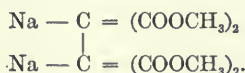
CONDENSED BENZENES AND THEIR DERIVATIVES

WITH the derivatives of the terpenes we close those benzene derivatives which are of interest to pharmacy, and are prepared to take up a new series of the hydrocarbons. These are found in the condensed benzenes—naphthalene and anthracene. Both of these substances are constituents of coal-tar, and are obtained by the fractional distillation of this product. The fraction distilling between 180° and 200° C. contains naphthalene, while that coming over from 340° to 360° C. contains anthracene.

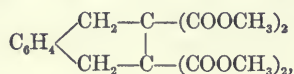
Naphthalene was discovered by Garden in 1816 in coal-tar. While naphthalene is always obtained commercially from coal-tar, its synthesis has been frequently accomplished. Thus, if methane, ethylene, and acetylene be passed through red-hot tubes, one of the products of this treatment is naphthalene. Other syntheses throwing light on chemical formula are matters of passing interest. Thus if the vapor of phenyl butylene dibromide,



be passed over red-hot lime, naphthalene results. This synthesis is of importance as showing that naphthalene contains a benzene nucleus. Still more important is the formation of naphthalene from combination of orthoxylenen bromide, $\text{C}_6\text{H}_4\begin{smallmatrix} \text{CH}_2\text{Br} \\ \text{CH}_2\text{Br} \end{smallmatrix}$, with the sodium salt of acetylene tetracarboxylic acid methyl ester,

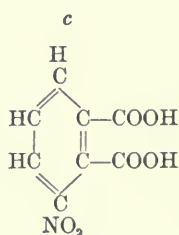
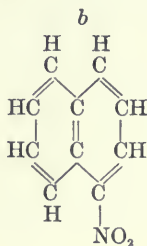
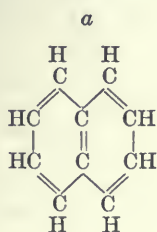


When they are brought together, we obtain hydronaphthalene-tetracarboxylic methyl ester,

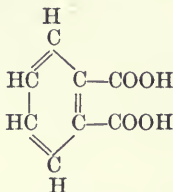


and this substance, with appropriate treatment, yields naphthalene. The importance of this synthesis is that it proves that naphthalene consists of the benzene group, attached to which is a ring containing four carbon atoms.

Two reactions of naphthalene are also of interest to us in helping to establish the formula. The first is that, on oxidation, it breaks up, yielding phthalic acid, $\text{C}_6\text{H}_4\begin{smallmatrix} \text{COOH} \\ \text{COOH} \end{smallmatrix}$; and the second is that, when treated with bromine, it easily absorbs four atoms, and then with difficulty absorbs six more. All these reactions point to the following graphic formula for naphthalene (a):



That it contains at least one benzene group is shown by the two syntheses given above. That the added ring containing four carbon atoms is in the ortho position to the benzene group is shown by the second synthesis given above, and also by the oxidation with phthalic acid. That the ring containing four carbon atoms has two double bonds is shown by the fact that it easily adds four hydrogen atoms; that the extra four carbon atoms are in a ring is shown by the behavior of its mononitro-derivative (b). Under certain treatment the pure benzene ring breaks, and we get nitrophthalic acid (c). Under other treatment it is the nitro ring that breaks, and we get pure phthalic acid,



NAPHTHALENUM. Naphthalene.

$$\text{C}_{10}\text{H}_8 = 127.10$$

[NAPHTALINUM, PHARM. 1890]

A hydrocarbon obtained from coal-tar, and purified by crystallization. It should be kept in well-stoppered, amber-colored bottles.

Colorless, shining, transparent laminae, having a strong, characteristic odor resembling that of coal-tar, and a burning, aromatic taste; slowly volatilized on exposure to air; by exposure to light acquiring a brownish color.

Insoluble in water, but when boiled with it, the water acquires a faint odor and taste; soluble in 13 parts of alcohol at 25° C. (77° F.), and very soluble in boiling alcohol; also very soluble in ether, chloroform, carbon disulphide, and fixed or volatile oils.

Naphthalene volatilizes slowly at ordinary temperatures, but rapidly when heated. It also volatilizes with the vapors of water or alcohol. At 80° C. (176° F.) it melts and at 218° C. (424.4° F.) it boils. Its vapor is inflammable, burning with a luminous but smoky flame. When ignited, it is consumed, leaving no residue.

Naphthalene is neutral to litmus-paper moistened with alcohol.

On shaking a small portion of Naphthalene with concentrated sulphuric acid, the acid should remain colorless, and should not acquire more than a pale reddish tint if the mixture be heated for five minutes on a water-bath (absence of *impurities derived from coal-tar*).

Tests for Identity.—Given above.

Impurities.—Coal-tar residues. Details above.

The above description is self-explanatory, the test that it should be colorless with sulphuric acid being a simple one to prove the absence of contaminations.

Naphthalene has been used considerably of late under the name of *coal-tar camphor*, and the popular form in which it is sold in the shape of small balls is called *moth-balls*. Its use in place of camphor is due to the increase in price of camphor which has taken place during the last decade, and not because of its greater value. It is used as an insecticide as is camphor, but there is no question as to preference of the aromatic odor of camphor to the rank, persistent odor of naphthalene. Naphthalene is also used as a carbureter of gas. Gas deficient in carbon, such as the so-called water-gas and natural gas, can be made to give a more luminous flame by passing the gas through cylinders or globes containing crystallized naphthalene. In the present day of incandescent burners the necessity of

the luminous flame is lessening, and so also is the application of naphthalene as a carbureter.

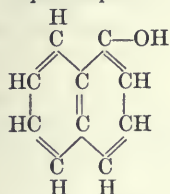
Naphthalene possesses antiseptic properties, and has been recommended as a remedy in chronic bronchitis and intestinal catarrh. Its chief use, however, is externally as an antiseptic dressing, and even for this purpose it is not very popular on account of its disagreeable odor.

Dose.—125 milligrammes (2 grains).

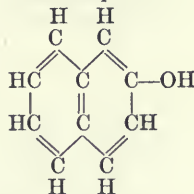
NAPHTHOLS

These are phenols of naphthalene. While there was but one phenol yielded from benzene,—phenol, or carbolic acid, U. S. P.,—there are two phenols from naphthalene; for, as shown in the following graphic formula, there is a difference whether the hydroxyl group is placed on the carbon atom next to the pure benzene ring or on the second carbon atom from the same. When placed on the first carbon atom, the derivative is called alpha-naphthol, whereas if placed on the second carbon atom, it is called beta-naphthol. What is true as far as alpha- and beta-naphthol are concerned is true in regard to all the mono-derivatives of the naphthols; two of each derivatives, alpha and beta, being possible.

Alpha-naphthol.



Beta-naphthol.



Both naphthols are found in coal-tar, and both can be made synthetically by treatment of naphthalene with concentrated sulphuric acid, when the naphthalene-sulphuric acid is produced. If the temperature is carefully kept at 160° C., the product will be the beta-derivative, no alpha-naphthene-sulphuric acid being produced. If this beta-derivative is fused with sodium hydroxide, betanaphthol results.

BETANAPHTHOL. Betanaphthol.

$$\text{C}_{10}\text{H}_7\text{OH} = 142.98$$

[NAPHTOL, PHARM. 1890]

A monatomic phenol occurring in coal-tar, but usually prepared from naphthalene. Betanaphthol should be kept in dark amber-colored, well-stoppered bottles.

Colorless or pale bluish-colored, shining crystalline laminae or a white or yellowish-white crystalline powder, having a faint phenol-like odor and a sharp and pungent but not persistent taste. Permanent in the air.

Soluble in about 950 parts of water and in 0.61 part of alcohol at 25° C. (77° F.); in about 75 parts of boiling water and very soluble in boiling alcohol; easily soluble in ether, chloroform, or solutions of alkali hydroxides.

Betanaphthol melts at 122° C. (251.6° F.) and boils at 286° C. (546.8° F.). It sublimes readily when heated; when in alcoholic or aqueous solution, it is volatilized with the vapor of alcohol or water. On ignition it leaves no residue.

It is neutral to litmus-paper which has been moistened with alcohol.

A cold saturated aqueous solution of Betanaphthol when mixed with ammonia water exhibits a faint bluish fluorescence.

On adding about 0.1 Gm. of Betanaphthol to about 5 Cc. of an aqueous solution of potassium hydroxide (1 in 4), then about 1 Cc. of chloroform, and gently warming the mixture, the aqueous layer will acquire a blue tint, changing afterward to green and brown.

Ferric chloride T.S. colors an aqueous solution greenish, and after some time causes the separation of white flakes, which turn brown upon the application of heat.

Betanaphthol should dissolve in 50 parts of ammonia water without leaving a residue (absence of *naphthalene*), and the solution should not have a deeper color than pale yellow (absence of *other organic impurities*).

An aqueous solution of Betanaphthol on the addition of chlorinated lime should show a pale yellow color and not a dark violet (distinction from, and absence of, *alphanaphthol*).

An aqueous solution of Betanaphthol, on the addition of a few drops of iodine T.S., followed by sodium hydroxide T.S. in excess, should show no color (distinction from, and absence of, *alphanaphthol*, which produces an intensely violet color).

Tests for Identity.—Given above.

Impurities.—Naphthalene, organic impurities, alphanaphthol. Details above.

Remarks.—Betanaphthol is used as an antiseptic both internally and externally, a solution of 1 : 3000 being a good germicide. It is given internally in intestinal troubles, especially in typhoid fever.

Dose.—250 milligrammes (4 grains).

Naphthol itself possesses rather irritating properties, and has a decidedly peppery taste. For that reason its application is somewhat limited, and many efforts have been made to prepare derivatives that are equally valuable as antiseptics and possessing less irritating properties. Among these derivatives are found the following:

Betol is naphthol salol or naphthol salicylate, $C_{10}H_7OOC C_6H_4OH$, and is made by treatment of the betanaphthol sodium compound with salicylic acid, in the presence of phosphorus oxychloride. The action of betol is similar to salol, except that when it enters the intestines it decomposes into naphthol instead of phenol.

Betol is given in doses of 1 Gm.

Alphanaphthol.—This naphthol derivative is said to act as a germicide in the proportion of 1 : 4000, and is also said to be one-third as poisonous as is betanaphthol.

Dose.—1 Gm.

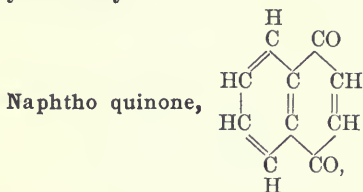
Benzonaphthol.—This is betanaphthol benzoate, and is, therefore, the benzoic acid ester of naphthol, and is prepared by combining naphthol with benzoyl chloride. It is a valuable intestinal antiseptic, and given in 1-Gm. doses.

Naphtholaristol.—Di-iodo-betanaphthol. This is an iodine derivative of naphthol, and is used as an antiseptic, under the name *iodonaphthol*.

Hydonaphthol.—This is a proprietary naphthol derivative, said to be betanaphthol, but this statement is denied by the manufacturer, who claims it is a product of the reduction of betanaphthol.

Asaprol.—This is a calcium betanaphthol monosulphonate, $(C_{10}H_6<\overset{OH}{SO_2})_2Ca$. It is sometimes called *abrostol*, and is used as an antipyretic in 0.3 to 1 Gm. doses.

Alumnol is aluminum betanaphthol disulphonate; that is, the aluminum salt of the acid, $C_{10}H_5(SO_3H)_2$. It is used as an astringent and antiseptic, almost entirely externally.



is known, and yields several important

dye-stuffs. Among them may be cited *carminic acid* (p. 939).

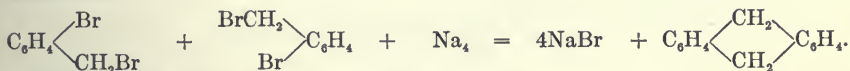
ANTHRACENE

Formula, $C_{14}H_{10}$. This substance, which was discovered by Dumas and Lamant in 1835, is of importance in pharmacy only because of its medicinal derivatives. The substance itself is found in coal-tar, and is obtained from same by fractional distillation. It can also be made by passing simple organic substances, such as methane, ethylene, or alcohol, through red-hot tubes.

Three syntheses of anthracene are worthy of mention as establishing the structural formula of the body. The first synthesis is its production, by treatment of orthotolyl-phenyl-ketone, with zinc-dust. This reaction is best shown by the following graphic equation:

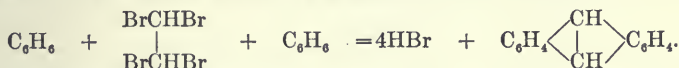


It can also be made from orthobrombenzyl bromide, treating same in ethereal solution with sodium, thus removing the bromine, with the formation of hydro-anthracene:



And this, when treated with an oxidizing agent, yields anthracene.

A third synthesis of anthracene is by treating benzene with tetrabromomethane in the presence of aluminum chloride, as is shown in the equation given below:



It may be recalled that naphthalene, when treated with nascent hydrogen, easily added four atoms of hydrogen, and with difficulty six more were persuaded to go on. Anthracene, likewise, easily adds four atoms of hydrogen, and under additional treatment it can be made to take up ten extra atoms of hydrogen, this seeming to point to the existence of seven double bonds, two of which are more easily replaceable than the other five.

The structure of anthracene is deduced from the following considerations:

First, it must possess two benzene rings, this being proved by the synthesis of orthotolylphenyl ketone and by the hydroanthracene synthesis.

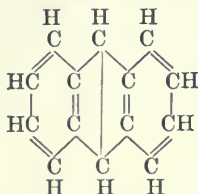
Secondly, the middle ring proceeds from the ortho position of both benzenes; this being proved by the synthesis with orthobrombenzyl bromide.

Thirdly, the middle ring has an ethylene bond extending between its two individual carbon atoms; this is shown by the hydro-anthracene synthesis.

Fourthly, this ethylene bond is weaker than the others, as shown by the ease with which the two hydrogens are taken away.

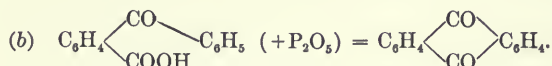
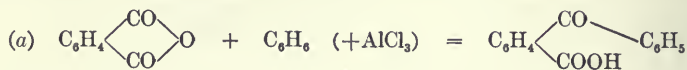
Fifth, that each of the two benzene rings have three ethylene bonds; this being proved by the fact that the body absorbs ten other atoms of hydrogen, but that with more difficulty than did the first two.

From these facts the characteristic formula of anthracene, shown below, is deduced:

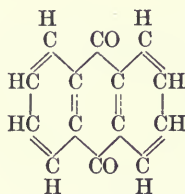


Anthraquinone.—Formula, $C_{14}H_8O_2$. This is an anthracene derivative in which two hydrogen atoms have been replaced by two oxy-

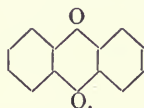
gen atoms. It is made by the treatment of phthalic acid anhydride with benzene in the presence of aluminum chloride, this yielding orthobenzoyl benzoic acid, which substance is converted into anthraquinone when treated with phosphorus pentoxide:



This reaction led to the conclusion that the graphic formula of anthraquinone is

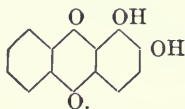


or, schematically,



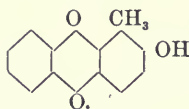
Anthraquinone can also be made by the oxidation of anthracene with chromic acid.

Anthraquinone is of little importance in pharmacy, but the oxyderivatives of it are worth more than passing notice. Among these we find *alizarine*, which is *dioxyanthraquinone*:



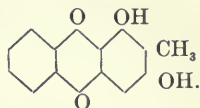
This substance, which is a coloring constituent of madder, was isolated from that dye-stuff by Graebe and Lieberman in the year 1867, while in 1879 they accomplished its synthesis by treating phthalic acid anhydride with pyrocatechin. *Madder* is the ground root of *Achusa tinctoria*, and formerly one of the staple dyeing materials was an infusion of this substance, which yielded, in the presence of aluminum salts, characteristic scarlet dyes. The madder industry represented an important item in the agriculture of southern France until the discovery of synthetic alizarine, since which time the industry has been practically ruined, as is shown in the following figures concerning the exportation of madder from France. In 1872 the exports of madder were valued at \$1,850,000; in 1878, after the discovery of artificial alizarine, the exports were only \$70,000; and in 1883 the exports have sunk to the insignificant figure of \$53,000.

Oxy-methylantraquinone,



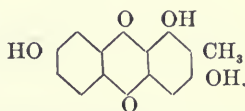
This substance was discovered by Tschirch in his valuable researches on the active principles of the well-known purgative resins, and considered by him as the fundamental purgative principle.

Dioxymethylantraquinone, or *chrysophanic acid*,



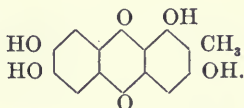
This substance represents an active purgative principle of chrysa-robin, senna, rhubarb, cascara sagrada. Note that this is not the chrysophanic acid of commerce, that name being erroneously applied to the natural product which the pharmacopœia calls chrysarobin (p. 873).

Trioxymethylantraquinone, or *emodin*,

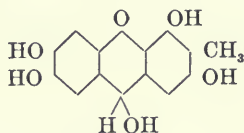


This is one of the important purgative principles found in rhubarb, frangula, and aloes.

Tetraoxymethylantraquinone,



Tetraoxymethylantraquinone is called *aloexanthin*, being one of the less important constituents of aloes. Aloin, according to Tschirch, is a body having the following graphic formula:



DRUGS CONTAINING THE ANTHRAQUINONE PURGATIVES

RHEUM. Rhubarb.

The dried rhizome of *Rheum officinale* Baillon, *Rheum palmatum* Linné, and the var. *tanguticum* Maximowicz (Fam. *Polygonaceæ*), or probably other species of *Rheum*, grown in China and Thibet, and deprived of most of the bark and carefully dried.

Constituents.—Chrysophanic acid; emodin; *chrysophan* (a glucoside that hydrolyzes to chrysophanic acid); resins (*phaeoretin*, *erythroretin*, etc.); rheotannic acid; starch, etc.

Remarks.—Rhubarb, one of our most valuable medicines, is the sliced and dried root of *Rheum officinale*. Study its constituents very carefully, noting that, besides the cathartic anthraquinone derivatives, it contains an astringent,—rheotannic acid,—and, therefore, while the drug, administered alone, has a purgative action, when combined with aromatics it acts as an astringent. This explains why aromatic syrup of rhubarb checks diarrhea, while syrup of rhubarb is a laxative.

Torrefied rhubarb (p. 84) is the roasted drug. The tannin is thus destroyed, and the pure purgative results.

Medical Properties.—Cathartic and astringent (see above). Official preparations: *Fluidextract* (p. 246), *tincture* (p. 235), *aromatic tincture* (p. 235), *compound powder* (p. 305), and *compound pills*. The *extract* (p. 289), the *syrup* (p. 198), and *mixture of rhubarb and soda* (p. 265) are made from the fluidextract, while the *aromatic syrup* (p. 198) is made from the aromatic tincture.

Dose.—Of drug, 1 Gm. (15 grains).

FRANGULA. Frangula. Buckthorn Bark.

The dried bark of *Rhamnus Frangula* Linné (Fam. *Rhamnaceæ*), collected at least one year before being used.

Constituents.—*Rhamnoxanthin* (or *frangulin*), which has been proved a mixture of *frangulinic acid* and *emodin*; tannin, bitter resin, etc.

Medical Properties.—Cathartic. Administered in the form of the official *fluidextract* (p. 247).

Dose.—1 Gm. (15 grains).

RHAMNUS PURSHIANA. Cascara Sagrada.

The dried bark of *Rhamnus Purshiana* De Candolle (Fam. *Rhamnaceæ*), collected at least one year before being used.

Constituents.—Chrysophanic acid, *emodin*, bitter resins, etc.

Remarks.—Cascara sagrada is one of our most popular purgatives. Its chief drawback is its bitter taste, but it has been found that the bitterness resides largely in a resinous principle, and can be removed by treatment with alkali, without seriously lessening the purgative action of the drug. This fact is applied in the manufacture of the official aromatic fluidextract, where the bark is freed from bitterness by treatment with magnesia.

Both frangula and cascara sagrada in fresh condition contain a ferment which causes griping, and which is dissociated on allowing the bark to age; hence the pharmacopœial requirement that both barks be at least one year old when used. As the pharmacist can never tell whether the fluidextract purchased in the open market has been made from the green bark, the only safe course is to make his own fluidextract from bark that he has kept in stock for one year. It is claimed, however, that heating the bark for a few hours at 100° C. destroys the ferment, and that barks can, therefore, be aged in this way.

Medical Properties.—Purgative of great service. There is, however, danger of contracting the "cascara habit." The *extract* (p. 288), *fluidextract* (p. 247), and the *aromatic fluidextract* (p. 254) are official.

Dose.—1 Gm. (15 grains).

SENNA. Senna.

The dried leaflets of *Cassia acutifolia* Delile (Alexandria Senna), or of *Cassia angustifolia* Vahl (India Senna) (Fam. *Leguminosæ*).

Constituents.—Cathartic acid (a glucoside which hydrolyzes to cathartogenic acid); chrysophanic acid; emodin; *chrysoretin* (a form of chrysophan), a bitter, griping resin.

Remarks.—Senna represents the leaflets of two plants, *Cassia acutifolia* being the source of the drug shipped from Egypt, and *Cassia angustifolia* yielding that exported from India. *Tinnevely Senna* is a fine variety coming from India. Strong alcohol removes the griping, resinous principle without extracting the true purgative constituents, cathartic acid, chrysophanic acid, and emodin. (See the Fluidextract, p. 255.)

Medical Properties.—Cathartic. Official preparations: The *fluid-extract* (p. 255), *compound infusion* (p. 215), and the *confection* (p. 313). *syrup of senna* (p. 201) is made from the fluidextract.

Dose.—Of drug, 4 Gm. (60 grains).

ALOE. Aloes.

[ALOE BARBADENSIS, ALOE SOCOTRINA, PHARM. 1890].

The inspissated juice of the leaves of *Aloe vera* (Linné) Webb, *Aloe Chinensis* Baker, *Aloe Perryi* Baker, or other species of *Aloe* (Fam. *Liliaceæ*).

Constituents.—Aloin, emodin, resin, etc.

Remarks.—It will be noted that the official definition given above mentions three sources of aloes. The plant *Aloes vera* furnishes Barbadoes aloes (*Aloes Barbadensis*, U. S. P. 1890), *Aloe Perryi* yields Socotrine aloes (*Aloe Socotrina*, U. S. P. 1890), while *Aloe Chinensis* produces what is called in commerce Curacao aloes. The aloin from these three varieties is not identical.

In passing it might be stated that these plants have huge succulent leaves, like those of the century plant, and that the drug is obtained by cutting these leaves from their base, allowing the juice to drain (in poorer varieties the leaves are expressed), and permitting it to dry in the sun.

Cape aloes is obtained from *Aloe Spicata* or *Aloe ferox*, and is now quite scarce. The so-called Cape aloes of commerce is of the poorest grade, and is unfit for human consumption.

Medical Properties.—Laxative, purgative, emmenagogue. Official preparations: *Purified aloes* (see below) and the *extract* (p. 279). Aloes should not be used in crude form, hence all official preparations save the extract are made from purified aloes.

Dose.—250 milligrammes (4 grains).

ALOE PURIFICATA. Purified Aloes.

Recipe.—Aloes, one thousand grammes..... 1000 Gm.
Alcohol, two hundred cubic centimeters..... 200 Cc.

Heat the Aloes, by means of a water-bath, until it is completely melted. Then add the Alcohol, and, having stirred the mixture thoroughly, strain it through a No. 60 sieve, which has just been dipped into boiling water. Evaporate the strained mixture by means of a water-bath, constantly stirring, until a thread of the mass becomes brittle on cooling. Lastly, break the product, when cold, into pieces of a convenient size, and keep it in well-stoppered bottles.

Purified Aloes is irregular, brittle pieces of a dull-brown or reddish-brown color, and having the peculiar odor of Aloes. It is almost entirely soluble in alcohol.

Remarks.—From above recipe it will be seen that aloes is purified by mixing with enough alcohol to make a liquid that can be strained through a No. 60 sieve, and that the strained liquid is then freed from alcohol by evaporation on a water-bath. In other words, the process is purely mechanical, the object being to free crude aloes from dirt, straw, and other insoluble impurities.

The official preparations made from purified aloes are the *tincture* (p. 221), *tincture of aloes and myrrh* (p. 221), the *pills* (p. 322), *pills of aloes and iron* (p. 322), *pills of aloes and mastic* (p. 322), and *pills of aloes and myrrh* (p. 323). It is also a constituent of *compound extract of colocynth* (p. 282).

Medical Properties and Dose.—Same as aloes.

ALOINUM. Aloin.

A neutral principle obtained from Aloes, varying more or less in chemical composition and physical properties according to the source from which it is obtained. Chiefly prepared from Curaçao Aloes.

A micro-crystalline powder or minute acicular crystals, lemon-yellow to dark yellow in color, possessing a slight odor of aloes and an intensely bitter taste. It is slightly hygroscopic, the air-dried powder yielding 1 molecule of water of crystallization at 100° C. (212° F.).

Aloin from Curaçao Aloes is soluble in about 120 parts of water, 15 parts of alcohol, 664 parts of ether, 4260 parts of chloroform, 3120 parts of benzene, 1475 parts of petroleum benzin, and 55 parts of acetone, at 25° C. (77° F.).

The melting-point of Aloin from Curaçao Aloes which has been dried over sulphuric acid, is about 147° C. (296.6° F.). When ignited, it is consumed without leaving a residue.

Aloin dissolves in water, forming a yellow solution, which is neutral to litmus-paper, turns brown on standing, and has an intensely bitter taste.

Ammonia water and alkali solutions dissolve Aloin, forming a yellow solution, soon turning red and exhibiting a greenish-red fluorescence.

Nitric Acid dissolves Aloin from Curaçao Aloes, forming a cherry-red solution (distinction from *Nataloin*, *Socaloin*, and *Capaloin*).

Aloin when added to alkaline solutions is rapidly decomposed, but if mixed with acid solutions decomposition is slower. Aloin added in minute quantity to concentrated sulphuric acid forms a yellowish-red solution which, upon the addition of a small crystal of potassium dichromate, changes to an olive-green, then to a dark green, and finally, on standing, to a blue. If a large amount of potassium dichromate be used, the yellowish-red solution first turns purple, then brown, and finally green. Bromine water added to the aqueous solution of Aloin produces a pink color. Gold chloride T.S., when added to an aqueous solution of Aloin, turns it carmine-red, changing later to violet. A drop of ferric chloride T.S. added to an alcoholic solution of Aloin produces a brownish-green color.

If to a dilute aqueous solution of Aloin, obtained from Curaçao Aloes, 1 drop of copper sulphate T.S. be added, a bright yellow color will be produced; upon adding a few drops of a concentrated solution of sodium chloride, the liquid will acquire a red color, and upon the further addition of a little alcohol the color will be changed to violet (distinction from *Nataloin* and *Capaloin*).

On shaking 1 Gm. of Aloin with 10 Cc. of benzene for one minute and filtering, the filtrate should not impart more than a faint pink color to an equal volume of ammonia water (5 per cent.) when shaken with it (limit of *emodin*).

Tests for Identity.—Given above.

Impurities.—*Nataloin*, *capaloin*, *socaloin*, and *emodin*. Details above.

Composition.—The chemistry of this body has been already discussed (p. 869).

Manufacture.—Aloes is treated with acidulated hot water, and

the infusion allowed to stand a day; the clear liquid is decanted from the sediment, concentrated at low heat, and set in a cool place, when the aloin will separate in minute crystals. This crude aloin is then purified by recrystallization from hot water. It is also made by treating an acid infusion of aloes with alkali and solution of calcium chloride. A combination of aloin with calcium is effected, and this separates out, the calcium being eventually removed by solution in diluted hydrochloric acid. The mixture is then treated with hot water, in which the resulting calcium chloride is very soluble, while the less soluble aloin crystallizes from the solution on cooling.

Remarks.—It will be noticed from the above tests that aloin gives a number of striking color reactions. Of these, the most important is the nitric acid test (Histed's test), since each of the different kinds of aloin gives different play of color when treated with this acid. Thus the official aloin (from Curaçao aloes) turns cherry red.

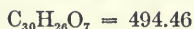
Nataloin (that from Natal aloes) turns vivid crimson.

Socaloin (that from Socotrine aloes) is not colored, while *capaloin* (that from Cape aloes) turns a permanent green.

Medical Properties.—Purgative. A constituent of *compound laxative pills* U. S. P. (p. 324).

Dose.—15 milligrammes ($\frac{1}{4}$ grain).

CHRYSAROBINUM. Chrysarobin.



A neutral principle extracted from Goa Powder, a substance found deposited in the wood of *Vouacapoua Araroba* (Aguiar) Druce (Fam. *Leguminosæ*). Chrysarobin should be preserved in amber-colored, glass-stoppered vials.

A pale orange-yellow, micro-crystalline powder, tasteless, odorless, and irritating to the mucous membrane.

Specific gravity: 0.920 to 0.922.

Soluble in 4812 parts of water, 308 parts of alcohol, 25 parts of benzene, 18 parts of chloroform, 114 parts of ether, 30 parts of amyl alcohol, and 230 parts of carbon disulphide at 25° C. (77° F.); soluble in 2170 parts of water at 80° C. (176° F.) and in 275 parts of alcohol at 60° C. (140° F.); soluble in dilute or concentrated solutions of potassium hydroxide, forming a red-colored liquid, with green fluorescence.

Chrysarobin, when heated, contracts and forms a dark mass at 148° C. (298.4° F.) and melts at 157° C. (314.6° F.). When ignited it is partly sublimed and finally entirely consumed.

In sulphuric acid it dissolves, forming a deep red solution. On pouring this liquid into water, Chrysarobin is deposited unchanged.

On mixing 0.001 Gm. of Chrysarobin with 2 drops of fuming nitric acid, a red-colored mixture is produced, which turns violet on the addition of a few drops of ammonia water (distinction from *chrysophanic acid*, which produces a yellow-colored liquid).

On shaking Chrysarobin with lime water for a few minutes, the liquid acquires a violet color (distinction from *chrysophanic acid*, which produces a yellow-colored liquid).

On adding a crystal of potassium dichromate to a solution of Chrysarobin in sulphuric acid, the red color is changed at first to green, then to purple, and finally to brown.

Tests for Identity.—Given above.

Impurities.—Chrysophanic acid. Details above.

Extraction.—Chrysarobin is obtained from Goa powder by extraction with hot chloroform, evaporation of the chloroformic solution to dryness, and powdering the residue.

Composition.—A reduction product of chrysophanic acid.

Remarks.—*Chrysarobin*, long and improperly called *chrysophanic acid*, is a derivative of Goa powder, a waste-product of the tree *Vouacapoua Araroba*. While excellent for skin diseases, chrysarobin is objectionable by reason of the stain it produces.

Medical Properties.—Drastic purgative. Generally used externally in skin troubles in the form of the official *ointment* (p. 345).

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

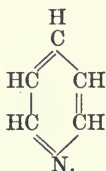
CHAPTER L

THE PYRIDINE AND QUINOLINE DERIVATIVES

It may be recalled (p. 752), that we have already considered a nitrogen compound, pyrrol, having the formula C_4H_4N . Pyridine is a ring compound similar to pyrrol, but having the formula C_5H_5N . It occurs in coal-tar, and has the disagreeable odor of bone oil.

It can be made by treating piperidine with concentrated sulphuric acid at a temperature of $300^\circ C$., piperidine, $C_5H_{11}N$, being made by heating pentamethylene-diamine hydrochloride. It can also be made from quinoline.

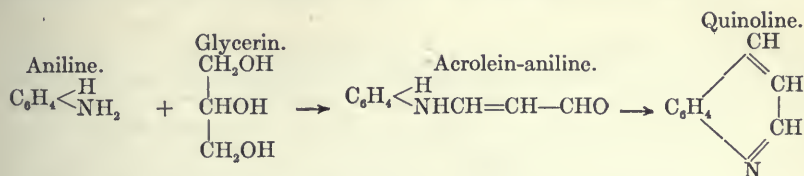
Pyridine resembles benzene in being oxidized with difficulty, and having the power to absorb six atoms of hydrogen or of any halogen. From this behavior and other reactions it is assumed that the formula of pyridine resembles that of benzene, except that in place of one of the carbon atoms in the benzene ring an atom of nitrogen is substituted, giving the graphic formula—



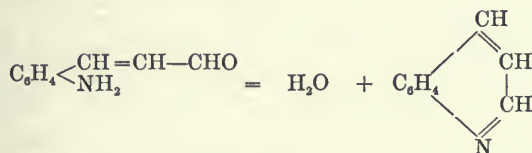
Pyridine is a colorless liquid, which, on account of its nauseating taste, is used for denaturizing alcohol—*i. e.*, for adding to alcohol which is to be used in the arts—and is, therefore, permitted to be sold without the revenue tax. The addition of the disagreeable constituent, pyridine, renders the use of such alcohol impossible as a beverage, and therefore accomplishes the purpose desired by the government. Pyridine is also used in asthma by inhalation, and is sometimes administered internally in doses of 2 to 10 minims.

Quinoline.—Formula, C_9H_7N . This substance, which is closely allied to pyridine, occurs, like it, in coal-tar and also in bone oil.

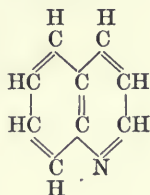
Quinoline is made by Skraup's reaction, which consists of treating aniline with glycerin in the presence of sulphuric acid and nitrobenzene.



It can also be made by dehydrating ortho-amido-cinnamic aldehyde.



On oxidation, quinoline yields pyridine dicarbonic acid, thus showing that it has a pyridine ring in its structure. It absorbs hydrogen in a way which indicates that not only does it possess a pyridine ring, but also a benzene ring, and to the substance is, therefore, assigned the following graphic formula:



Besides the methods of synthesis of quinoline given above, the chemical has been produced by the fusion of quinine with potassium hydroxide, and it was by this means that the compound was discovered and received its name.

ALKALOIDS

Alkaloids are an important class of plant bases having complex composition; in fact, some chemists considering the phrase "alkaloid" as covering only those basic organic substances whose chemistry is not clearly defined. This statement, while extreme, is partially justified by the fact that, as alkaloidal bodies are studied, they are frequently assigned to other divisions of chemistry. Thus *betaine* from beets is now grouped among the derivatives of glyocol, or amidoacetic acid, CH_2NH_2 (p. 673); choline from bile is classed as a



derivative of tetramethyl ammonium hydrate, $\text{N} \equiv (\text{CH}_3)_4$ (p. 633); while caffeine is now considered as a uric-acid derivative (p. 749), the pharmacopœia dropping the word "alkaloid" in discussing it, calling it "a feebly basic substance." There still remains, however, a distinct class of bodies which we can call alkaloids; the new definition

for alkaloids being *nitrogenous plant bases, which are derivatives of pyridine, quinoline, or similar ringed bodies.*

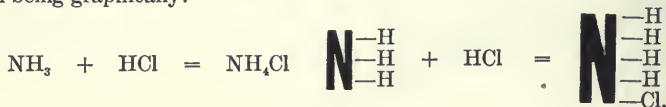
Hence it is necessary to drop the old classification of alkaloids and turn to the new, but before so doing it is advisable, from the historic point of view, to discuss a few phases of the old alkaloidal classification still dear to the present generation of pharmacists, at the same time calling attention to the fallacies found in each statement, which in most cases consist in the fact that while the statement is true for alkaloids it also applies to other substances no longer considered alkaloids.

I. "Alkaloids are basic, turning red litmus-paper blue, and combining with acids to form salts." True, but so do other bodies, such as methylamine (p. 648) and aniline (p. 767).

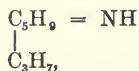
II. "Alkaloids are nitrogen bases, and are built on the ammonia type." True, but so are such compounds as mono-, di-, and tri-methylamine, and tetramethyl ammonium iodide (p. 648).

Before leaving this subject, however, it is well to employ it in order to explain why the formula of any alkaloidal salt is the formula of the alkaloid from which derived, plus the formula of the acid, *including its hydrogen*. Thus the formula of morphine hydrochloride, U. S. P. VIII., is $C_{17}H_{19}NO_3HCl$, while the formula of morphine, U. S. P. VIII., is $C_{17}H_{19}NO_3$.

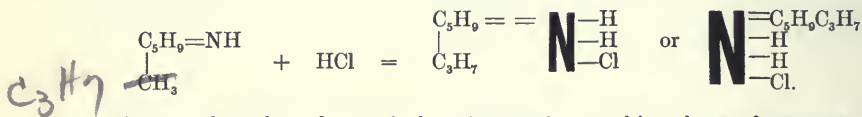
We have already learned (p. 483) the formation of a salt from ammonia was different from the formation of a sodium, potassium, or any other metallic salt, the reaction being graphically:



On this ammonia type are the alkaloids built, one, two, or all the hydrogen atoms being replaced by complex organic radicles; thus, one of the simplest, coniine, is—



and just as $NH_3 + HCl = NH_4Cl$, as shown above, so does coniine plus hydrochloric acid combine as follows:



Even so does the valence of the nitrogen in morphine change from III to V in the conversion of the free alkaloid into the salt. This also explains why the salt is named morphine hydrochloride (hydrochlorate, U. S. P. 1890), and not morphine chloride.

III. "There are a special line of reagents producing precipitates with alkaloids. Such alkaloidal precipitants are Mayer's reagent (potassio-mercuric iodide), gold chloride, tannic acid, etc." True, but these same reagents precipitate almost any amido derivative.

IV. "Alkaloids are grouped into the amines (liquid alkaloids) and the amides (solid alkaloids)." By amines we mean those bodies having the general formula RNH_2 (p. 633), while amides are those bodies having the general formula $RCOCH_3$ —that is, where the hydroxyl of the carboxyl group is replaced by the NH_2 group. The statement that the solid alkaloids are amides is not wholly true, as the solid alkaloids, atropine, cocaine, and morphine, do not contain the $RCOCH_3$ group.

Alkaloids are either solid or liquid, the greater number of them being solid. Among the liquid alkaloids that might be mentioned are nicotine, coniine, and lobeline. No fixed rule can be given as to the difference in the structure between the solid and the liquid alka-

loids; the general statement that the liquid alkaloids are free from oxygen is not always true. Thus, the liquid alkaloid pelletierine contains oxygen.

Alkaloids usually occur in the form of salts, the most abundant natural alkaloidal combinations being citrates, malates, and tannates. The alkaloids found in cinchona are generally salts of quinic acid, while the alkaloids of opium are usually combined in the drug with meconic acid. So far, very few alkaloids have been found in monocotyledonous plants, the only two of importance derived from plants of that order being colchicine and veratrine, the greater majority of the alkaloids being found in the plants of the dicotyledonous series, although curious to relate, two of the most important families of this group, the two containing the largest number of species, namely, the natural orders Compositæ and Labiata, contain no alkaloids. On the other hand, with some plant families, such as the Solanaceæ and Papaveraceæ, almost every represented species contains alkaloids.

As to nomenclature, note that the pharmacopœia reserves the termination "ine" for alkaloids only, using "in" for glucosides and neutral principles. Other countries do not make this distinction, terminating the names of all active principles of drugs with "in." For comparison, let us tabulate the pharmacopœial Latin and English names of a typical glucoside and a typical alkaloid:

	LATIN.	ENGLISH.
GLUCOSIDE, U. S. P. VIII.....	Salicinum.	Salicin.
ALKALOID, U. S. P. VIII.....	Quinina.	Quinine.
ALKALOID, U. S. P. 1870.....	Quinia.	

The last termination has still considerable hold in the Southern States, where there are often calls for "sulphate of cinchonidia" (now cinchonidina in Latin, and cinchonidine in English) or for "quinia sulphas."

EXTRACTION OF ALKALOIDS

While the extraction of each alkaloid from its drug means a difference in manipulation according to the peculiarities of the individual alkaloid, and while an outline of the separation of each alkaloid is given under the proper heading, a general idea of alkaloidal extraction can here be outlined.

The drug is extracted with acidulated water or alcohol. If the latter menstruum is used, the alcohol is distilled off and the residue taken up with acid, and this acidulated aqueous liquid is made alkaline with potassa,—soda, ammonia, or milk of lime,—thereby liberating the alkaloid which precipitates from solution. The freed alkaloid is either collected on a filter and purified by recrystallization from the appropriate solvent or more generally while still in the liquid is "shaken out" (p. 137) with a solvent immiscible in water—say gasoline, petroleum ether, chloroform, or ether. The freed alkaloid dissolves in this solvent, the solution separated from the now exhausted aqueous layer, the solvents evaporated off, and the crude alkaloid purified by recrystallization, usually as a salt rather than as the free alkaloid. Of course, this bare outline omits the details of purification employed in each case, these details being taken up in discussing the individual drugs.

CLASSIFICATION OF ALKALOIDS

Pyridine derivatives:

Piperidine.	
Piperine.	
Coniine.	Nicotine.
Pilocarpine, U. S. P.	Sparteine, U. S. P.

Tropine derivatives:

Tropine.	Hyoscine, U. S. P.
Atropine, U. S. P.	Hyoscyamine, U. S. P.
Scopolamine, U. S. P.	Cocaine, U. S. P.

Quinoline derivatives:

Quinine, U. S. P.	Strychnine, U. S. P.
Quinidine.	Veratrine, U. S. P.
Cinchonine, U. S. P.	
Cinchonidine, U. S. P.	

Isoquinoline derivatives:

Isoquinoline.	Hydrastine, U. S. P.
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Phenanthrene derivatives:

Morphine, U. S. P.	Codeine, U. S. P.
Apomorphine, U. S. P.	

Unallotted:

Physostigmine, U. S. P.	Pelletierine, U. S. P.
Aconitine, U. S. P.	
Colchicine, U. S. P.	Cornutine.

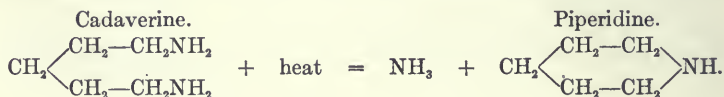
The other alkaloids, such as *emetine*, *gelsemine*, *cissampeline*, etc., are but little understood.

Note.—In the table just given, alkaloids to which the initials “U. S. P.” are appended are official either as free alkaloid or as a salt. In other words, it does not necessarily mean that the free alkaloid is official, nor that none of its salts is official.

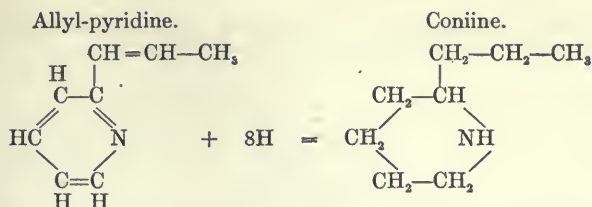
GROUP I.—THE PYRIDINE DERIVATIVES

The chemistry of pyridine has already been considered on p. 874. Its hydrogen compound, *hexahydropyridine*, $C_5H_{11}N$, is called *piperidine*, and was discovered by Rochleder in 1849 on heating piperine with soda lime.

Piperidine is obtained from *cadaverine* (or *pentamethylene diamine*), a substance found in decaying flesh. On heating this body ammonia is driven off, with the formation of piperidine, as shown below:



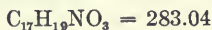
Piperidine is here mentioned not because of its value in pharmacy, but because its propyl derivative is an alkaloid of importance by reason of being the first alkaloid made synthetically. This alkaloid is coniine. *Coniine* was isolated by Giesecke in 1827 from the fruit of *Conium maculatum*. It is alpha-propyl-piperidine, and its synthesis was accomplished by Ladenberg in 1886 by reduction of allyl-pyridine:



Synthetic coniine is identical with the natural product obtained from conium, except that it is optically inactive, while the natural coniine is dextrogyrate. As already mentioned on p. 685, when an inactive form of an optically active chemical is known, it usually consists of equal quantities of the dextrogyrate and the lævogyrate forms of this substance. Following this theory, the inactive synthetic coniine must be a mixture of the dextrogyrate and the lævogyrate alkaloid. Now, if the natural coniine is the dextrogyre, while the artificial is a mixture of the dextrogyre with lævogyre, it follows that if lævogyre is removed, the dextrogyre will remain, and by reason of this theory an effort was made to remove the lævogyrate form from the synthetic inactive product by special crystallizations, and a product exactly identical with the natural was obtained.

As mentioned above, coniine is the alkaloid found in *Conium maculatum*, and is rarely used in medicine.

PIPERINA. Piperine.



A feebly basic substance $[\text{CH}_2\text{O}_2\cdot\text{C}_6\text{H}_3\cdot\text{CH}:\text{CH}:\text{CH}:\text{CON}\cdot\text{C}_5\text{H}_{10}]$ obtained from Pepper and other plants of the *Piperaceæ*.

Colorless or pale yellowish, glistening, monoclinic crystals, odorless, permanent in the air, and containing no water of crystallization. When put into the mouth it is at first tasteless, but on prolonged contact it develops a sharp, biting taste. It is optically inactive.

Insoluble in water; soluble in 15 parts of alcohol, 36 parts of ether, and in 1.7 parts of chloroform at 25° C. (77° F.); soluble in 4.4 parts of alcohol at 60° C. (140° F.).

It melts at 130° C. (266° F.). Upon ignition, it emits alkaline vapors, and is completely consumed.

Its alcoholic solution is neutral to litmus-paper.

Sulphuric acid dissolves Piperine with the formation of a blood-red color, which disappears on dilution with water.

On heating 0.1 Gm. of Piperine with alcoholic potassium hydroxide, it is converted into piperinic acid and piperidine, the latter recognizable by its alkaline, pepper-like odor, and the former by its melting-point, 215° C. (419° F.).

On adding a crystal of Piperine to sulphuric acid containing about half its volume of solution of formaldehyde, a permanently green liquid is formed.

On adding a crystal of Piperine to sulphuric acid containing a fragment of potassium dichromate, it at once acquires a purple color, and, on stirring, dissolves, forming a reddish-brown solution, which becomes greenish on adding water to it.

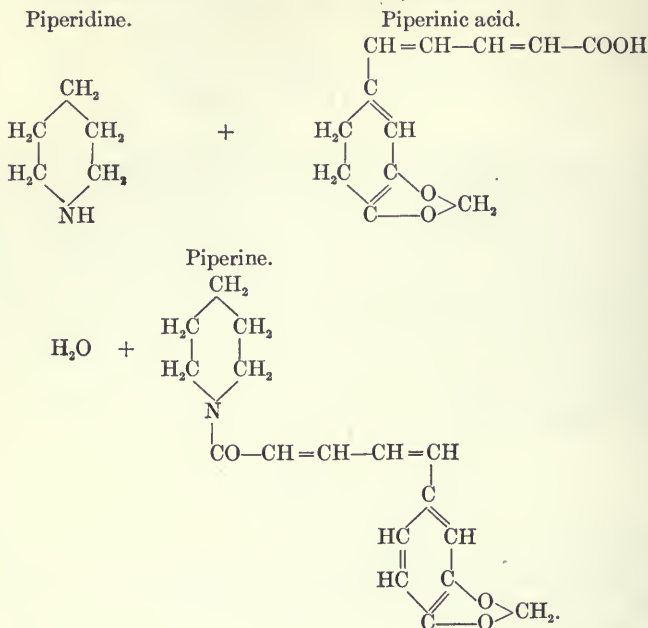
On adding a crystal of Piperine to sulphuric acid containing a trace of selenous acid, it turns brown, changing at once to violet, and dissolves, forming a brown solution, changing to green.

When heated with nitric acid, Piperine is colored at first orange, then red, and the acid acquires a yellow color, deepening to reddish as the crystals dissolve. On adding to this solution an excess of potassium hydroxide T.S., the color is at first yellow, but upon boiling it becomes blood-red.

Tests for Identity.—Given above.

Extraction.—The alcohol extract of white pepper is freed from resin by treatment with solution of potassa, and after removing the resin solution by washing with water, the insoluble residue, consisting chiefly of piperine, is dissolved in alcohol and crystallized several times from the same solvent.

Constitution.—Piperine is the combination of piperidine with piperinic acid, and has been made synthetically from these substances, viz.:



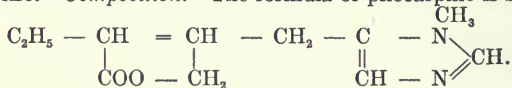
Likewise, when treated with alcoholic potassa, piperine splits into piperidine and piperinic acid, this dissociation being included among the pharmacopœial tests.

As piperine does not readily form salts with acids, some authorities no longer consider it an alkaloid, the pharmacopœia seeming to hold to that opinion by defining it as “a feebly basic substance.”

Piperine is a useful antiperiodic.

Dose.—200 milligrammes (3 grains).

Pilocarpine.—*Composition.*—The formula of pilocarpine is supposed to be—



Extraction.—Jaborandi in fine powder is moistened with sodium carbonate solution and then extracted with warm benzene. The benzene percolate containing the free alkaloid is shaken with diluted hydrochloric acid (which dissolves the alkaloid as hydrochloride), and the separated acid liquid is made alkaline with sodium carbonate solution and then shaken out with chloroform. The chloroform solution of the free alkaloid is next evaporated, the crude alkaloid taken up with diluted nitric acid, and the nitrate thus purified by recrystallization from alcohol.

The free alkaloid can be obtained from the nitrate by making its aqueous solution alkaline with ammonia, shaking out with chloroform, and evaporation of the chloroform.

The “shaking out” with chloroform will be explained in Part V., under Extraction with Immiscible Solvents.

The following salts of pilocarpine are official.

PILOCARPINÆ HYDROCHLORIDUM. Pilocarpine Hydrochloride.

[PILOCARPINÆ HYDROCHLORAS, PHARM. 1890]

The hydrochloride [$HCl \cdot C_{11}H_{16}N_2O_2$] of an alkaloid obtained from *Pilocarpus*. It should be kept in well-stoppered, amber-colored vials.

Colorless or white, transparent crystals, odorless, and having a faintly bitter taste; deliquescent on exposure to the air. It contains no water of crystallization.

Soluble in 0.3 part of water, 2.3 parts of alcohol, and in 540 parts of chloroform at 25° C. (77° F.); soluble in 1.1 part of alcohol at 60° C. (140° F.); insoluble in ether.

After drying for several hours at 100° C. (212° F.) the salt melts at 195.9° C. (384.5° F.), and upon ignition it is entirely consumed.

Its aqueous solution is neutral or has a faintly acid reaction upon blue litmus-paper.

Sulphuric acid dissolves Pilocarpine Hydrochloride, with elimination of hydrochloric acid gas and the formation of a colorless liquid; upon adding to the solution a small fragment of potassium dichromate on a white porcelain surface, a bright, grass-green color is produced.

If 0.01 to 0.02 Gm. of the salt be dissolved in 2 Cc. of water in a test-tube, 2 Cc. of a slightly acid solution of hydrogen dioxide added, and a small layer of benzene be carefully poured upon the liquid, then if 3 or 4 drops of a solution of potassium dichromate (1 in 300) be added and the mixture be gently shaken, the benzene layer will acquire a violet color, while the aqueous layer will remain yellow (distinction from other alkaloids). If more than 0.02 Gm. be used, the benzene turns blue, and the reaction is no longer characteristic).

On rubbing together equal parts of Pilocarpine Hydrochloride and mercurous chloride, a black color is produced.

Tests for Identity.—For pilocarpine, see above. For chlorides, see Part V.

Remarks.—The color produced with sulphuric acid and potassium dichromate is quite characteristic, each alkaloid giving a different shade with the combination. In the case of pilocarpine a grass-green color is produced. The salts of pilocarpine, like the drug *pilocarpus*, are valuable sudorifics and sialagogues.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

PILOCARPINÆ NITRAS. Pilocarpine Nitrate.

The nitrate [$NO_2OH \cdot C_{11}H_{16}N_2O_2$] of an alkaloid obtained from *Pilocarpus*. It should be kept in well-stoppered, amber-colored vials.

Colorless or white, shining crystals, odorless, and having a faintly bitter taste; permanent in the air. It contains no water of crystallization.

Soluble in 4 parts of water and in 60 parts of alcohol at 25° C. (77° F.); soluble in 16 parts of alcohol at 60° C. (140° F.); insoluble in ether and chloroform.

It melts at 170.9° C. (339.7° F.). Upon ignition, it is quickly consumed, leaving no residue.

The aqueous solution (1 in 100) shows an acid reaction to blue litmus-paper.

Sulphuric acid dissolves Pilocarpine Nitrate, forming a colorless solution, and on adding a fragment of potassium dichromate, a bright grass-green color is produced.

On rubbing together equal parts of the salt and mercurous chloride, no black color is produced (distinction from *pilocarpine hydrochloride*).

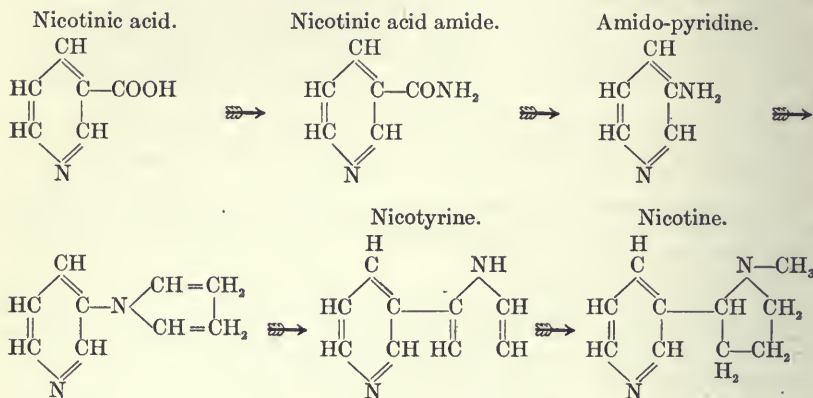
Tests for Identity.—Physical tests given above. For pilocarpine, see the hydrochloride. For nitrates, details in Part V.

Impurities.—Pilocarpine hydrochloride. Details above. Other alkaloids. See the hydrochloride.

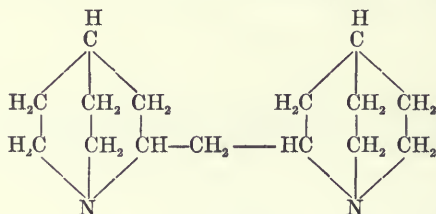
Medical Properties.—Used just like the hydrochloride.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

Nicotine.—Nicotine was isolated from tobacco by Posselt in 1828. Investigations show that this alkaloid is piperidyl-tetrahydro-methyl-pyrrol, the graphic formula of which is given below. The structural formula was worked out by Pictet and Crepieux in 1895, whose work, however, did not lead to the synthesis of the alkaloid, but only as far as nicotyrin, the derivative of nicotine shown in the graphic formulas given below:



Sparteine.—The structural formula of this alkaloid is supposed to be:



SPARTEINÆ SULPHAS. Sparteine Sulphate.



The sulphate $[\text{SO}_4(\text{OH})_2 \cdot \text{C}_{15}\text{H}_{26}\text{N}_2 + 5\text{H}_2\text{O}]$ of an alkaloid obtained from *Scoparius*. Sparteine Sulphate should be kept in well-stoppered, amber-colored vials.

Colorless, rhombohedral crystals, or a crystalline powder; odorless and having a slightly saline and somewhat bitter taste. It crystallizes with varying amounts of water of crystallization, but when recrystallized from a solution in diluted alcohol it contains 5 molecules of water.

Soluble in 1.1 parts of water and in 2.4 parts of alcohol at 25° C. (77° F.); insoluble in ether and chloroform.

When heated to 110° C. (230° F.) the salt loses all of its water of crystallization (21.3 per cent.), and when anhydrous, melts at 136° C. (276.8° F.).

It is hygroscopic, and its aqueous solution has an acid reaction upon blue litmus-paper.

If 25 Cc. of ether be added to about 0.1 Gm. of Sparteine Sulphate in a test-tube, followed by a few drops of diluted ammonia water, avoiding an excess, and if an ethereal solution of iodine (1 in 50) be afterward added until the liquid, when shaken, turns from an orange to a dark reddish-brown color, the bottom and sides of the test-tube will, after a short time, be coated with minute, dark greenish brown crystals.

If a solution of potassium ferrocyanide (1 in 20) be added to a solution of the salt, a yellow precipitate is produced.

Sulphuric acid, when added to Sparteine Sulphate, should not become colored (absence of sugar and other readily carbonizable organic impurities).

On shaking 0.05 Gm. of Sparteine Sulphate, in a test-tube, with 5 Cc. of potas-

sium hydroxide T.S., the liquid will at first be turbid, and small drops of Sparteine will gradually collect on the surface. If a strip of moistened red litmus-paper be suspended in the mouth of the test-tube, and a gentle heat then applied, the test paper will gradually acquire a blue color, but no odor of ammonia should be perceptible (absence of ammonium salts).

Tests for Identity.—For sparteine, given above. For sulphates, details in Part V.

Impurities.—Sugar, etc.; ammonium salts. Details above.

Extraction.—The drug is extracted with acidulated water, the liquid made alkaline with sodium hydroxide, and from this alkaline liquid the alkaloid, which is volatile, is separated by distillation. It is purified by dissolving in diluted hydrochloric acid, evaporated to dryness, and the alkaloid freed by treatment with potassium hydroxide is redistilled. The salts are made from the alkaloid by treatment with the appropriate acid.

Remarks.—Sparteine sulphate is a valuable diuretic.

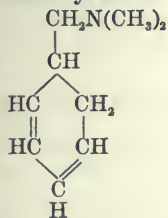
Dose.—10 milligrammes ($\frac{1}{3}$ grain).

GROUP II.—THE ATROPINE DERIVATIVES

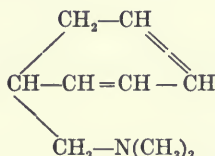
The very important alkaloids of the nightshade families are derivatives of tropine, this being an artificial alkaloid which is a decomposition product of atropine, and since its structure is similar to that substance from which it is derived, chemists consider it as the base upon which atropine is built.

The synthesis of tropine is shown in the successive series of graphic formulas given below:

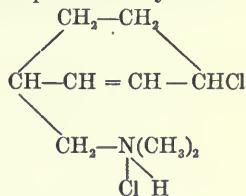
Dihydrobenzyl-
dimethyl-amine.



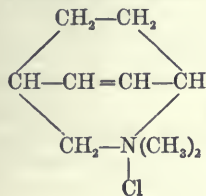
or



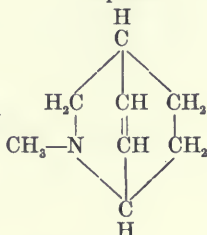
Hydrochloride of methyl
tropine chlorhydrate.



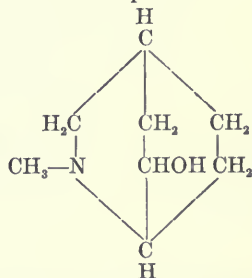
Tropidine methyl
ammonium chloride.



Tropidine.

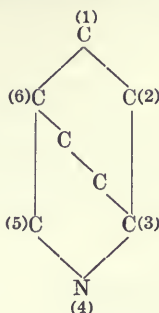


Tropine.



It will be seen that the synthesis starts with a compound consisting of a benzene ring with a side chain containing an amido group, and terminates with a ring similar to the pyridine structure, with a carbon chain in the center. The tropine ring differs

from the pyridine ring in the position of the nitrogen atom. Or, to express it differently, the tropin formula consists of a pyridine ring with the carbon chain extending diagonally from carbon atom 3 to carbon atom 6.



ATROPINA. Atropine.

$$C_{17}H_{23}NO_3 = 287.04$$

An alkaloid obtained from *Atropa Belladonna* Linné (Fam. *Solanaceæ*) and from other plants of the same family. As it occurs in commerce, it is usually accompanied by a small amount of hyoscyamine, from which it cannot be readily separated. It should be kept in amber-colored, well-stoppered vials.

White rhombic prisms, more or less elongated in the direction of the major axis as they contain more or less hyoscyamine; odorless; possessing a bitter, acid taste (*it should be tasted with the utmost caution and only in dilute solution*). Atropine shows an alkaline reaction with litmus, phenolphthalein, and hematoxylin T.S.

Pure Atropine is optically inactive, being composed of Dextro-Atropine (Dextro = +10.02°) and Lævo-Atropine (Lævo = -9.22°), both of whose melting-points are 111°C. (231.8° F.), and the melting-point of whose chloraurates are both 146° C. (294.8° F.). The chloraurate may be made by adding gold chloride T.S. to a solution of Atropine in diluted hydrochloric acid, washing, collecting, and drying the precipitate.

Pure atropine chloraurate melts at 136° C. (276.8° F.).

Pure hyoscyamine chloraurate melts at 160° C. (320° F.).

Pure hyoscyne chloraurate melts at 197° C. (386.6° F.). Atropine chloraurate alone melts under boiling water.

At about 113.8° C. (237° F.) Atropine melts, forming a colorless liquid; the melting-point of Atropine free from hyoscyamine is about 115.8° C. (240.4° F.). When ignited it is consumed without leaving a residue.

Soluble in 450 parts of water, 1.46 parts of alcohol, 16.6 parts of ether, and 1.56 parts of chloroform at 25° C. (77° F.); soluble in 86.7 parts of water at 80° C. (176° F.) and in 0.9 part of alcohol at 60° C. (140° F.).

On adding sulphuric acid to Atropine, no color should be produced (absence of readily carbonizable organic impurities), nor should any color be developed upon the subsequent addition of nitric acid (absence of, and difference from, *morphine*).

In a solution of Atropine in hydrochloric acid, platinic chloride T.S. produces no precipitate (difference from *most other alkaloids*). Gold chloride T.S. yields a yellow, lustreless precipitate in such a solution.

If a few crystals of Atropine be placed in a porcelain dish on a water-bath with a few drops of nitric acid, and heated to dryness, a yellow residue will be produced; if on cooling a few drops of alcoholic potassium hydroxide T.S. and a fragment of potassium hydroxide be added, an intense violet color is produced; hyoscyamine and hyoscyne will produce the same color, but the presence of strychnine masks the reaction.

If a crystal of Atropine be added to a few drops of sulphuric acid containing 1 drop of creosol, a pink color should be produced, which is not dissipated by the addition of 0.5 Gm. of hydrated chloral (distinction from *other alkaloids*, *hyoscyamine* producing a brown color and *strychnine* a black, *hyoscyne* remaining colorless).

If a small quantity of Atropine, or one of its salts, be heated with a few Cc. of sulphuric acid, a peculiar odor, recalling that of a mixture of rose, orange flower,

and melilot, will become noticeable. The addition of a few fragments of potassium dichromate will change this odor to that of bitter almond.

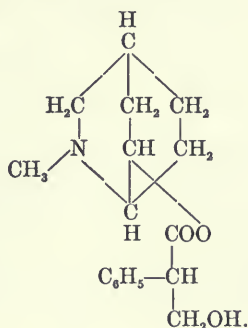
Tests for Identity.—Given above.

Impurities.—Carbonizable organic matter, morphine, other alkaloids. Details above.

Extraction.—The alcoholic percolate from belladonna is made alkaline by treatment with milk of lime and then filtered. The filtrate (a solution of free alkaloid) is acidulated with diluted sulphuric acid, freed from fat and resin by evaporation of the alcohol, and taking up with acidulated water. The acidulated aqueous filtrate is then treated with alkali, and the free alkaloid thus precipitated is washed with water, dissolved in alcohol, and crystallized from this solution by addition of a little water.

Remarks.—Atropine is a tropine tropate, and can be made by treating tropine with tropic acid in the presence of hydrochloric acid.

Tropic acid is phenyl-hydracrylic acid, $\text{C}_6\text{H}_5\text{—CH} \begin{matrix} \text{CH}_2\text{OH} \\ \text{COOH} \end{matrix}$ and when it is combined with tropine, the product of the union—atropine—is assumed to have the following formula:



It will be noticed from the reactions given above that potassium dichromate and sulphuric acid give with atropine not only a green color, but also a benzoic-like odor. Vitali's reaction for atropine is interesting. When the alkaloid is treated with fuming nitric acid and evaporated to dryness, and a drop of potassium hydroxide be added to the dry residue, it will give a violet-blue color, and if the residue is again covered with alcohol, the isonitril odor (p. 642) is evolved.

Atropine is a sedative and mydriatic.

Dose.— $\frac{4}{100}$ milligramme ($\frac{1}{1600}$ grain).

ATROPINÆ SULPHAS. Atropine Sulphate.

$$(\text{C}_{17}\text{H}_{23}\text{NO}_3)_2 \cdot \text{H}_2\text{SO}_4 = 671.43$$

The sulphate $[\text{SO}_2(\text{OH})_2 \cdot (\text{C}_{17}\text{H}_{23}\text{NO}_3)_2]$ of an alkaloid obtained from *Atropa Belladonna* Linné (Fam. *Solanaceae*), and from other plants of the same family. As it occurs in commerce, it usually contains small amount of hyoscyamine sulphate, from which it cannot be readily separated.

A white crystalline powder or microscopical needles and prisms (the form of the latter being probably due to the hyoscyamine present); odorless, having a very bitter, nauseating taste, and permanent in the air. *It should be tasted with the utmost caution and only in dilute solution.*

Soluble in 0.38 part of water, 3.7 parts of alcohol, 2140 parts of ether, and in 620 parts of chloroform at 25° C. (77° F.); soluble in 0.22 part of water at 80° C. (176° F.) and in 1.9 parts of alcohol at 60° C. (140° F.).

At about 189.9° C. (373.5° F.) Atropine Sulphate melts; when free from hyoscyamine it melts at about 188° C. (370.4° F.). When ignited it chars, emits acrid vapors, and is rapidly and completely consumed.

On adding potassium hydroxide T.S. to a solution of Atropine Sulphate, a white precipitate of atropine is obtained, which should respond to the reactions and tests given under *Atropina*.

Tests for Identity.—Given above.

Remarks.—Atropine sulphate, being more soluble than the uncombined alkaloid, is generally used instead of the latter.

Dose.— $\frac{4}{16}$ milligramme ($\frac{1}{16}$ grain).

HOMATROPINÆ HYDROBROMIDUM. Homatropine Hydrobromide.



The hydrobromide $[HBr \cdot C_{16}H_{21}NO_3]$ of an alkaloid obtained by the condensation of tropine and mandelic acid. It should be kept in well-stoppered vials, protected from light.

A white, odorless, crystalline powder, or rhombic prisms, having a bitter taste.

Soluble in 5.7 parts of water, 32.5 parts of alcohol, and in 620 parts of chloroform at 25° C. (77° F.); soluble in 8.7 parts of alcohol at 60° C. (140° F.); insoluble in ether.

At 213.8° C. (417° F.) it melts. It leaves no residue upon incineration.

Its aqueous solution is neutral to litmus-paper, and is not precipitated by tannic acid T.S. or platinic chloride T.S. It contains no water of crystallization.

If 2 Cc. of chloroform be shaken with 1 Cc. of an aqueous solution of the salt (1 in 10) to which a few drops of chlorine water have been cautiously added, the chloroform should assume a brownish color.

Iodine T.S., when added to solutions of Homatropine Hydrobromide, produces a brown precipitate; silver nitrate T.S., a creamy white precipitate.

If to an aqueous solution containing 0.1 Gm. of the salt an excess of potassium hydroxide T.S. be added, this liquid shaken out with ether, and the ether allowed to evaporate spontaneously, the crystals which form should have a melting-point of 96° C. (204.8° F.).

If 1 Cc. of an aqueous solution of the salt (1 in 100) be made alkaline with ammonia water, shaken out with chloroform, and the chloroformic solution evaporated to dryness, the residue should turn yellow and finally brick-red when warmed with about 1.5 Cc. of a solution made by dissolving 1 part of mercuric chloride in 50 parts of a mixture of alcohol 5 volumes and water 3 volumes (absence of *most other alkaloids except atropine and hyoscyamine*).

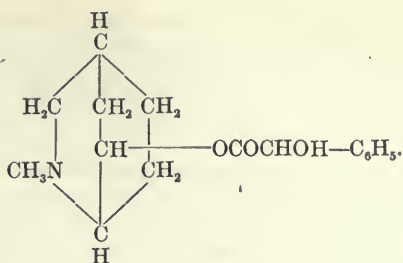
If sulphuric acid containing a trace of potassium dichromate be added to a crystal of the salt, an evanescent pink color will be produced, which changes rapidly to green.

If 0.01 Gm. of the salt be added to 5 drops of nitric acid, and evaporated to dryness in a porcelain dish, the residue should not acquire a violet color upon the addition of a few drops of alcoholic potassium hydroxide T.S. (absence of *atropine, hyoscyamine, or hyoscyne*).

Tests for Identity.—Given above.

Impurities.—Other alkaloids, atropine, hyoscyamine, and hyoscyne. Details above.

Remarks.—Homatropine is an artificial alkaloid made by the condensation of tropine and mandelic acid. It is phenyl-glycocoltropin.



Its action is similar to atropine, but is less poisonous. As a mydriatic—a dilator of the pupil of the eye—it is preferable to atropine, because it works more rapidly.

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

Hyoscyamine.—Hyoscyamine, as the formula $C_{17}H_{23}NO_3$ shows, is isomeric with atropine, and when either atropine or hyoscyamine is treated with weak baryta water, both split into tropic acid and tropine. When it is said that hyoscyamine and atropine are isomeric, it must not be understood that the two are identical, for we have already learned, when considering tartaric acids (p. 684), that there may be many chemicals having similar formula, but different characteristics.

Recent investigations show that atropine (which is optically inactive) consists of the official hyoscyamine (which is lævogyre) and a dextrogyrate alkaloid isomeric with hyoscyamine.

Extraction.—Hyoscyamine is extracted either from the mother liquor left after atropine crystallization or from hyoscyamus seeds freed from fat by percolation with petroleum ether. The fat-free seeds are extracted with 85 per cent. alcohol acidulated with hydrochloric acid, the alcohol evaporated from percolate in vacuo, and the watery residue again treated with petroleum ether to remove fat and color. The liquid is then made alkaline with potassium carbonate and shaken out with chloroform. The chloroformic solution is then evaporated at low temperature, and the crude alkaloid that remains is purified by crystallization from diluted sulphuric acid solution. From this crystalline sulphate the alkaloid can be obtained by making its aqueous solution alkaline, shaking out with chloroform, and evaporating the chloroformic solution.

The pharmacopœia provides in the gold chloride test the method of distinguishing the hyoscyamine from atropine. Hyoscyamine, like atropine, is not precipitated by platinic chloride test solution, as are most of the alkaloids. Free hyoscyamine is not official, but its hydrobromide and sulphate are.

HYOSCYAMINÆ HYDROBROMIDUM. Hyoscyamine Hydrobromide.



[HYOSCYAMINÆ HYDROBROMAS, PHARM. 1890]

The hydrobromide [$HBr \cdot C_{17}H_{23}NO_3$] of an alkaloid obtained from *Hyoscyamus* and other plants of the *Solanaceæ*. It should be kept in amber-colored, well-stoppered vials.

White, prismatic crystals, or a yellowish, amorphous, resin-like mass, having, particularly when damp, a tobacco-like odor, and an acid, nauseous, and bitter taste; deliquescent on exposure to the air.

Very soluble in water; soluble in 2 parts of alcohol, 1600 parts of ether, and in 2.5 parts of chloroform at 25° C. (77° F.).

Its aqueous solution is neutral to litmus-paper and is strongly lævogyrate.

It melts at 151.8° C. (305.3° F.). It leaves no residue on incineration.

Gold chloride T.S., when added to a solution of the salt, yields a precipitate which, when recrystallized from a small quantity of boiling water acidulated with hydrochloric acid, is deposited, on cooling, in minute, lustrous, golden-yellow scales (difference from *atropine*).

Tests for Identity.—Physical tests given above.

Melting-point of chloraurate. See *Atropine*. Melting-point of atropine picrate, 175° C. Melting-point of hyoscyamine picrate, 162° C. Tests for bromide. Details in Part V.

Color test with nitric acid and alcoholic potassa. See *Atropine*.

Impurities.—*Atropine*. Details above. Other alkaloids, carbonizable matter, and morphine. For details see *Atropine*.

Extraction.—See under *Hyoscyamine*.

Remarks.—This salt has an effect similar to *atropine*.

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

HYOSCYAMINÆ SULPHAS. Hyoscyamine Sulphate.



The neutral sulphate $[\text{SO}_2(\text{OH})_2 \cdot (\text{C}_{17}\text{H}_{23}\text{NO}_3)_2]$ of an alkaloid obtained from *Hyoscyamus* and other plants of the *Solanaceæ*. It should be kept in amber-colored, well-stoppered vials.

White, indistinct crystals, or a white powder; odorless, having a bitter, acrid taste; deliquescent when exposed to the air.

Very soluble in water; soluble in 6.4 parts of alcohol, 2500 parts of ether, and in 2300 parts of chloroform at 25° C. (77° F.).

Its aqueous solution is lævogyrate and neutral to litmus-paper.

It melts at 198.9° C. (390.1° F.). Upon ignition it is rapidly consumed without residue.

Tests for Identity.—Physical tests. Given above. Tests for hyoscyamine. Details under the Hydrobromide. Test for sulphates. Details in Part V.

Impurities.—Same as those mentioned under the Hydrobromide.

Remarks.—The sulphate is used just as is the hydrobromide.

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

Hyoscine.—This alkaloid has the formula $\text{C}_{17}\text{H}_{21}\text{NO}_4$, and is isomeric with scopolamine (p. 889). Ten years since there was much controversy relative to the composition of hyoscine, some authorities claiming it was isomeric with atropine and hyoscyamine.

Hyoscine is made from the mother liquor remaining after removal of hyoscyamine from the seed of *hyoscyamus* or *stramonium*, by treating same with hydrobromic acid, and crystallizing the resultant hydrobromide.

The crude salt is purified by formation of the double chloride of the alkaloid and gold, and eventual separation of the gold by use of hydrogen sulphide.

HYOSCINÆ HYDROBROMIDUM. Hyoscine Hydrobromide.



[HYOSCINÆ HYDROBROMAS, PHARM. 1890]

The hydrobromide $[\text{HBr} \cdot \text{C}_{17}\text{H}_{21}\text{NO}_4 + 3\text{H}_2\text{O}]$ of an alkaloid, chemically identical with scopolamine, obtained from *Hyoscyamus* and other plants of the *Solanaceæ*. It should be kept in well-stoppered, amber-colored vials.

Colorless, transparent, rhombic crystals, sometimes of large size, odorless, having an acid, slightly bitter taste; slightly efflorescent.

Soluble in 1.5 parts of water, 16 parts of alcohol, and in 750 parts of chloroform at 25° C. (77° F.); soluble in 1.33 parts of alcohol at 60° C. (140° F.); insoluble in ether.

Its aqueous solution shows a slightly acid reaction to blue litmus-paper.

Hyoscyne Hydrobromide, when heated, softens at about 100° C. (212° F.); it first melts and then loses its water of crystallization at 110° C. (230° F.). If dried over sulphuric acid until deprived of its water of crystallization, it melts at 179.7° C. (355.5° F.). When ignited it leaves no residue.

If 0.01 Gm. of the salt be added to 5 drops of nitric acid, and evaporated to dryness in a porcelain dish, the residue should yield a violet color upon the addition of alcoholic potassium hydroxide T.S.

Mercuric potassium iodide T.S., in aqueous solutions of the salt acidified with hydrochloric acid, yields a yellowish-white precipitate.

Mercuric chloride T.S. and solution of phosphotungstic acid give a white precipitate when added to an aqueous solution of the salt.

Picric acid T.S. and platinic chloride T.S. yield yellow precipitates when added to a concentrated solution of the salt.

Tests for Identity.—Physical tests, given above. Melting-point of the chloraurate. See Atropine. Tests for hyoscyne. Given above. Tests for bromide. Details in Part V.

Impurities.—Carbonizable matter and morphine. For details, see Atropine.

Uses.—Similar to hyoscyamine.

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

SCOPOLAMINÆ HYDROBROMIDUM. Scopolamine Hydrobromide.

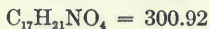


The hydrobromide [$HBr.C_{17}H_{21}NO_4 + 3H_2O$] of an alkaloid obtained from plants of the *Solanaceæ*; chemically identical with Hyoscyne Hydrobromide (see *Hyoscyne Hydrobromidum*).

Remarks.—As mentioned in the official definition, this body is identical with hyoscyne hydrobromide.

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

COCAINA. Cocaine.



An alkaloid [$C_8H_{13}(C_6H_5CO)NO.COOC_2H_5$] obtained from several varieties of Coca.

Large, colorless, four-sided or six-sided monoclinic prisms, having a slightly bitter taste, and producing on the tongue a temporary numbness.

Soluble in 600 parts of water, 5 parts of alcohol, and in 3.8 parts of ether at 25° C. (77° F.); very soluble in chloroform and warm alcohol; soluble in 260 parts of water at 80° C. (176° F.); soluble in benzene, carbon disulphide, ethyl acetate, in about 14 parts of oil of turpentine, and in about 12 parts of olive oil; insoluble in glycerin.

It melts at 98° C. (208.4° F.), and should leave no residue on ignition.

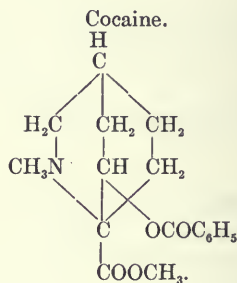
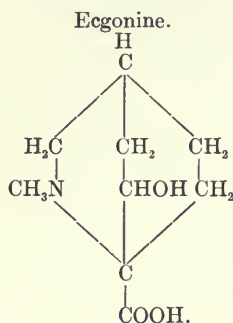
If an alcoholic solution of Cocaine be carefully neutralized with hydrochloric acid, and the solution evaporated to dryness, the residue should respond to the reactions and tests given under *Cocainæ Hydrochloridum*.

Tests for Identity.—Given above.

Extraction.—Coca leaves are extracted with 5 per cent. sulphuric acid, the percolate concentrated, made alkaline with sodium carbonate, and shaken out with kerosene. The kerosene solution is shaken with acidulated water, alkaloid precipitated from the aqueous liquid by addition of sodium carbonate, and the freed alkaloid

obtained by evaporation of the ethereal solution is purified by again dissolving in acidulated water, precipitation with alkali, and recrystallization from alcohol or ether.

Remarks.—This important alkaloid, which was first isolated from coca-leaves by Gaedeke in 1855, is now known to be benzoyl-methyl-ecgonine, the ecgonine being a carbonic acid derivative of tropine, a comparison of the formulas of the two substances, ecgonine and cocaine, being given below:



Dose.—30 milligrammes ($\frac{1}{2}$ grain). Used in Oleate of Cocaine (p. 211).

COCAINÆ HYDROCHLORIDUM. Cocaine Hydrochloride.

$$\text{C}_{17}\text{H}_{21}\text{NO}_4 \cdot \text{HCl} = 337.10$$

[COCAINÆ HYDROCHLORAS, PHARM. 1890]

The neutral hydrochloride $[\text{HCl} \cdot \text{C}_8\text{H}_{13}(\text{C}_6\text{H}_5\text{CO})\text{NO} \cdot \text{COOCH}_3]$ of an alkaloid obtained from several varieties of Coca.

Colorless, transparent monoclinic prisms, flaky, lustrous leaflets, or a white crystalline powder; permanent in the air, containing no water of crystallization; odorless; of a saline, slightly bitter taste, and producing on the tongue a tingling sensation followed by numbness of several minutes' duration.

Soluble in 0.4 part of water, 2.6 parts of alcohol, and in 18.5 parts of chloroform at 25°C . (77°F .); soluble in 0.1 part of water at 80°C . (176°F .), and in 1.4 part of alcohol at 60°C . (140°F .); insoluble in benzene, petroleum benzin, and ether.

It melts at about 189.9°C . (373.8°F .). Minute quantities of impurities may reduce the melting-point to 180°C . (356°F .) or less. It leaves no residue on incineration.

Its aqueous solution is neutral to litmus-paper and is lævogyrate.

On adding 5 drops of a solution of chromium trioxide (1 in 20) to 5 Cc. of a solution of Cocaine Hydrochloride (1 in 50), a yellow precipitate will be produced, which redissolves on shaking; on now adding 1 Cc. of hydrochloric acid, a permanent orange-colored, crystalline precipitate will be formed.

On adding a solution of potassium chromate (1 in 20) to a hydrochloric acid solution of the salt, orange-yellow leaflets of cocaine chromate are precipitated.

If mercuric chloride T.S. be added to an aqueous solution of the salt, a white flocculent precipitate is produced.

Cocaine Hydrochloride is not colored by cold sulphuric acid, but if a crystal be heated with sulphuric acid, in a test-tube, vapors are produced, from which, benzoic acid is deposited on cooling.

If three drops of palladous chloride T.S., with 3 Cc. of chlorine water, be added to 3 drops of an aqueous solution of the salt (1 in 20), a red precipitate is produced.

When 0.01 Gm. of the salt is dissolved in 2 drops of water, the addition of 1 Cc. of a solution of potassium permanganate (1 in 30) produces a violet precipitate, which appears brownish-violet when collected on a filter.

A crystal of the salt dissolved in alcohol yields, when stirred with a piece of potassium hydroxide, an odor of ethyl benzoate.

If 0.1 Gm. of the salt be dissolved in 5 Cc. of distilled water containing 3 drops of diluted sulphuric acid, the addition to this solution of 3 drops of tenth-normal potassium permanganate V.S. will produce a violet color, which should not fade in half an hour (limit of *cinnamyl-cocaine*).

If 0.1 Gm. of the salt be dissolved in 85 Cc. of cold distilled water, in a beaker, and 4 drops of ammonia water added, and the solution stirred vigorously for fifteen minutes, with occasional rubbing of the sides of the beaker with a stirring rod, a crystalline precipitate of cocaine should be formed, and the supernatant liquid should be perfectly clear (limit of *isatropyl-cocaine*). The presence of 0.5 per cent. of isatropyl-cocaine will prevent the formation of nearly all of the precipitate, and will cause the supernatant liquid to be opalescent.

Tests for Identity.—Physical tests. Given above. Tests for cocaine. Given above. Tests for chloride. Details in Part V.

Impurities.—Cinnamyl cocaine and isoatropyl cocaine. Details above.

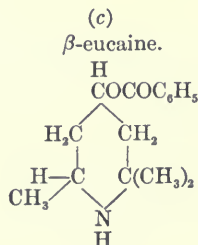
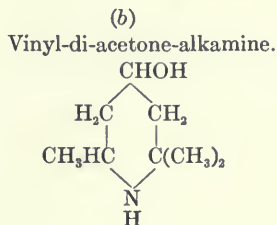
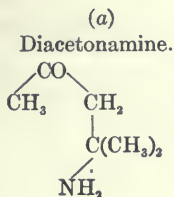
Remarks.—This soluble salt of cocaine is the one generally used whenever cocaine is indicated.

While cocaine was isolated as far back as 1855, the use of the alkaloid dates from the last twenty years, a demand for the product not being started until local anesthetic action of cocaine had become known in 1884. When small quantities of cocaine are rubbed on the surface or injected beneath the skin, a numbing of the nerves in that immediate neighborhood results, and so thorough is this insensibility that operations can be performed without pain where great agony would be produced were the parts not cocainized. On account of this valuable action of cocaine its use in all minor surgical operations spread widely, particularly in dentistry and operations on the nose and throat. From its original legitimate purpose cocaine has been prostituted into producing a sedative action in cases where wholly unnecessary. This is particularly true of catarrh powders, and it is through the use of such catarrh powders containing cocaine that the abuse of the drug increased throughout the country, until it has now become one of our national perils. It behooves the conscientious pharmacist, therefore, to do all in his power to stem this tide of cocaine abuse, for there are few drugs which act in a more rapidly degrading way than does cocaine.

Dose.—30 milligrammes ($\frac{1}{2}$ grain).

Eucaïne or beta-eucaïne is a synthetic alkaloid, the hydrochloride of trimethylbenzoyl-hydroxypiperidine.

It is made by treating diacetoneamine—(a) with paraldehyde, the product reduced by use of metallic sodium to vinyl-di-acetone-alkamine (b), and this treated with benzoyl chloride, whereupon the free base eucaïne (c) results.



It will be seen that eucaine has no chemical connection with cocaine, but, being used like it, is considered at this place. It is claimed that eucaine is an efficient local anesthetic, and that it does not produce a debasing habit, as does cocaine. Eucaine possesses the advantage over cocaine that it is stable and its solutions can be boiled, thus permitting sterilization. Cocaine when heated to the temperature of sterilization ($110^{\circ}\text{C}.$) is apt to decompose.

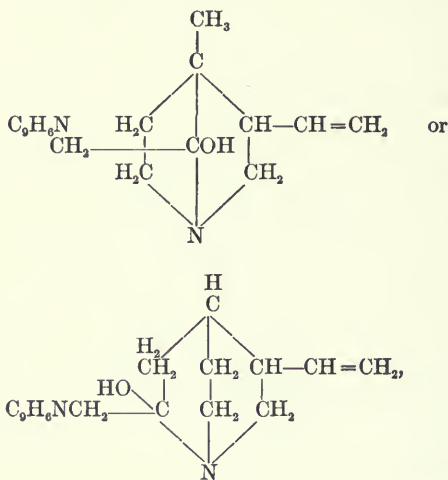
Dose.—Used only externally as local anesthetic.

Alpha-eucaine.—The hydrochloride of benzoyl-methyl-tetra-methyl-oxypiperidine-carboxylic-methyl-ester is used like beta-eucaine.

GROUP III.—QUINOLINE DERIVATIVES

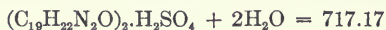
Quinoline itself has already been discussed on p. 875, where the structural formula is fully explained, and here reference need only be made to the alkaloid derivatives of this base.

Cinchonine ($\text{C}_9\text{H}_6\text{N}-\text{C}_9\text{H}_{12}\text{OHNCH}_3$).—Cinchonine is one of the three official alkaloids obtained from the cinchona bark. Of the species of cinchona yielding more or less of this alkaloid, it is found most abundantly in Huanuco cinchona. The structure of cinchonine has been studied from the decomposition products, and is supposed to be either—



the quinoline base being attached to a complex piperidine derivative.

CINCHONINÆ SULPHAS. Cinchonine Sulphate.



The neutral sulphate $[\text{SO}_2(\text{OH})_2 \cdot (\text{C}_{19}\text{H}_{22}\text{N}_2\text{O})_2 + 2\text{H}_2\text{O}]$ of an alkaloid obtained from the bark of several species of Cinchona.

White, hard, lustrous, prismatic crystals; odorless, permanent in the air, and having a bitter taste. At $100^{\circ}\text{C}.$ ($212^{\circ}\text{F}.$) it loses its water of crystallization. If crystallized from water it will contain two, and if from alcohol, one, molecule of water of crystallization. Its aqueous solution should not show fluorescence.

Soluble in 58 parts of water, 10 parts of alcohol, 2300 parts of ether, and in 69 parts of chloroform at $25^{\circ}\text{C}.$ ($77^{\circ}\text{F}.$); soluble in 32 parts of water at $80^{\circ}\text{C}.$ ($176^{\circ}\text{F}.$) and in 5.2 parts of alcohol at $60^{\circ}\text{C}.$ ($140^{\circ}\text{F}.$).

It melts at $198.5^{\circ}\text{C}.$ ($389.5^{\circ}\text{F}.$) and leaves no residue on incineration.

Its aqueous solution is neutral to litmus-paper and is dextrogyrate.

One Gm. of the salt dried to constant weight at 100° C. (212° F.) should weigh not less than 0.95 Gm. (absence of an *undue amount of water*).

A solution of the salt (1 in 1000) in diluted sulphuric acid should not exhibit more than a faint blue fluorescence (limit of *quinine* or *quinidine sulphates*).

If 1 part of the powdered salt be macerated with frequent agitation in 80 parts of chloroform, at ordinary temperatures, it should be wholly, or almost wholly, dissolved (limit of *quinine* or *cinchonidine sulphates*).

Tests for Identity.—Cinchonine. See above. Sulphates. Details in Part V.

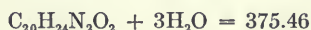
Impurities.—Excess water, quinine, quinidine, cinchonidine. Details above. Carbonizable matter—see Atropine.

Extraction.—Obtained from the mother liquor after the extraction of quinine (p. 896) and after the quinidine and cinchonidine have been removed by treatment explained on p. 900. The liquid freed from these three alkaloids is treated with soda, when the cinchonine precipitates as a resinous mass. This, on solution, in diluted sulphuric acid and purification by repeated crystallizations, yields cinchonine sulphate, which on treatment with alkali gives the free alkaloid.

Remarks.—Of the reactions for cinchonine, the only one calling for special attention is the interesting fact that, differing from quinine and cinchonine, it does not give a blue fluorescence in acid solutions. Cinchonine is used as an antipyretic—to reduce fevers—and as a remedy for malaria.

Dose.—250 milligrammes (4 grains).

QUININA. Quinine.



An alkaloid obtained from the bark of various species of *Cinchona* (Fam. *Rubiaceæ*). Quinine should be kept in well-stoppered, amber-colored bottles.

A white, flaky or micro-crystalline powder, odorless, having a bitter taste, and slightly efflorescent in dry air. Quinine, when freshly crystallized, should contain three molecules of water of crystallization. When heated, it fuses at 57° C. (134.6° F.), and loses two molecules of water of crystallization at 100° C. (212° F.), and the third molecule at 125° C. (257° F.); at ordinary temperatures the loss is gradual.

Quinine free from water is soluble in 1750 parts of water, 0.6 part of alcohol, 4.5 parts of ether, 1.9 part of chloroform, 158 parts of glycerin, 120 parts of benzene, 3450 parts of a solution of potassium hydroxide (1 in 20), and in 1810 parts of ammonia water at 25° C. (77° F.). It is soluble in 810 parts of water at 80° C. (176° F.).

Quinine containing three molecules of water is soluble in 1550 parts of water, 0.6 part of alcohol, 1.3 part of ether, 1.6 parts of chloroform, 212 parts of glycerin, 166 parts of benzene, 3450 parts of a solution of potassium hydroxide (1 in 20), and in 1810 parts of ammonia water at 25° C. (77° F.). It is soluble in 775 parts of water at 80° C. (176° F.).

When rendered anhydrous by heating to constant weight at 125° C. (257° F.) its melting-point is 174.9° C. (346.8° F.).

Its aqueous solution is levogyrate and is alkaline to moistened limus-paper.

If to 1 Cc. of an aqueous solution of quinine (1 in 100), containing just sufficient diluted sulphuric acid to effect complete solution, there be added 2 Cc. of bromine T.S., followed by 1 Cc. of ammonia water, the liquid should acquire an emerald-green color (*thalleioquin*).

If 0.7 Gm. of Quinine be dissolved in a mixture of 15 Cc. of acetic acid, 6 Cc. of alcohol, and 0.5 Cc. of sulphuric acid, the solution heated to boiling, and 7 Cc. of a saturated solution of iodine in alcohol be added slowly, bronze or olive-green crystals of quinine iodo-sulphate will separate on gradually cooling the solution. These crystals are insoluble in cold water.

If 0.2 Gm. of Quinine be dissolved in 1 Cc. of diluted sulphuric acid, the solution diluted with distilled water to 20 Cc. and neutralized with ammonia water, and 1 drop of solution of hydrogen dioxide and 1 drop of copper sulphate T.S. be added, and the liquid boiled, an intensely red color should appear, which slowly changes to a blue, and finally to a green color. (Quinine and quinidine alone respond to this test.)

One Gm. of Quinine should dissolve completely in a slightly warmed mixture of 6 Cc. of absolute alcohol and 3 Cc. of ether, which solution should remain clear on cooling (absence of *cinchonine* and *cinchonidine*).

Quinine should not impart more than a faintly yellowish tint to sulphuric acid (limit of *readily carbonizable organic impurities*), nor produce a red color with nitric acid (difference from *morphine*).

Quinine should not lose more than 14.3 per cent. of water on heating to 125° C. (257° F.) (absence of an excess of water).

When heated with 2 Cc. of potassium hydroxide T.S., no ammonia should be evolved (absence of *ammonium salts*).

Dissolve 2 Gm. of Quinine, which has previously been dried at 50° C. (122° F.) for two hours in a porcelain dish, in 20 Cc. of alcohol. Add 2 drops of hematoxylin T.S., neutralize exactly with sulphuric acid, and evaporate to dryness on a water-bath. Complete the test by following the directions given on page 899, under *Quinina Sulphas* (Test for Other Cinchona Alkaloids, Section II.).

Tests for Identity.—Given above.

Impurities.—Cinchonine, cinchonidine, excess of water. Carbonizable matter, nitric acid, and ammonia. Details above.

Remarks.—This important alkaloid was isolated by Pelletier and Caventou, two apothecaries of Paris, in 1820. Investigations show that quinine is a methoxycinchonine, $\text{CH}_3\text{O}-\text{C}_9\text{H}_7\text{N}-\text{C}_9\text{H}_{12}\text{OHNCH}_3$.

The synthesis of quinine has not yet been accomplished.

Quinine can be obtained from cinchona bark by the method given in the official assay of cinchona. (See p. 1018.) In this case the cinchona is treated with a mixture consisting of ether, chloroform, and ammonia water, the object of the latter being to free the alkaloids from their natural combinations with quinic acid, the separated alkaloids being dissolved by the mixture of ether and chloroform. This ethereal chloroformic solution of the freed alkaloids is then treated with normal sulphuric acid, converting the alkaloids into the sulphates, which then dissolve in the aqueous sulphuric acid layer. This aqueous liquid is then separated from the ether-chloroform layer and rendered alkaline with ammonia, when the alkaloids precipitate and are again taken up with a mixture of chloroform and ether. This chloroform-ether solution is then evaporated, when the alkaloids are left in more or less crystalline form and the quinine is separated from the other alkaloids by fractional crystallization.

The commercial extraction of quinine is accomplished by mixing the powdered bark with calcium hydroxide (milk of lime), thus setting the cinchona alkaloids free from their natural combination in the bark, and then extracting the freed alkaloids with gasoline. This gasoline solution is then shaken with diluted sulphuric acid, and the acidulated watery solution after separation from the gasoline is neutralized with sodium carbonate, after which, the liquid on crystallization, yields an impure quinine sulphate, which is purified by repeated crystallizations from a sulphuric acid solution neutralized with sodium carbonate. The free alkaloid is obtained from the sulphate by treatment of its acid solution with an alkali.

Of the reactions given above, quite characteristic is the blue

fluorescence produced when quinine is dissolved in acid. A very beautiful test is the thalleioquin test; which is given above, consisting of the treatment of a faintly acid quinine solution with bromine water, and the excess of ammonia, when the liquid assumes a vivid green tint.

The test given above, based on the formation of green crystals of quinine iodosulphate, is quite characteristic, the iodosulphate being sometimes called *herapathite*. A test distinguishing quinine and quinidine from cinchonine and cinchonidine is the one given above, based on change of color of a solution of the two alkaloids when treated with copper sulphate and hydrogen dioxide.

Besides, in the free form, quinine is recognized by the pharmacopœia as bisulphate, hydrobromide, hydrochloride, salicylate, and sulphate. Of these salts of quinine the most popular is the sulphate. Quinine sulphate is only sparingly soluble in water, but when an aqueous mixture of the sulphate is treated with sulphuric acid, the salt rapidly goes into solution. This is due to the formation of the bisulphate of quinine, one part of which dissolves in 10 parts of water, whereas one part of quinine sulphate requires 740 parts of water for solution. There is a question, however, as to the wisdom of dispensing quinine solutions, as by dissolving the salt its bitter taste is made still more pronounced. It is, in fact, preferable to administer the same in a mixture with a "shake well" label, and in such a mixture the taste of the quinine can be effectively masked by the use of preparations of licorice, such as ammoniated glycyrrhizin or an aqueous fluidextract of licorice (such as is now official). Compound syrup of sarsaparilla has also been found useful to mask the taste of quinine, but it is not so effective as are licorice preparations.

There has been a great demand for the preparations of quinine devoid of the disagreeable taste. For adults the administration of quinine capsules covers the need, but in the case of children who cannot swallow capsules some liquid method of administration is exceedingly desirable. The nearest approach to a palatable quinine liquid is a licorice combination of quinine, as cited above. There have been put on the market proprietary liquid preparations purporting to contain quinine, but investigation shows that such preparations contain practically no quinine, the less bitter and also less effective quinidine being used instead. Such a preparation is the syrup of quinidine of the National Formulary. In fact, practically the only way satisfactorily to administer quinine internally to children is as quinine tannate in the form of chocolate lozenges, and this method is highly satisfactory. The physiologic effect of quinine can also be secured by preparing an ointment of quinine (the alkaloid or the sulphate) with benzoinated lard or petrolatum and lanoline, this ointment being applied by rubbing under the armpits. Oleate of quinine, U. S. P., can be used for the same purpose.

Physicians at times prescribed quinine with licorice, then direct addition of diluted sulphuric acid to dissolve the quinine. In such combination (see p. 1077) the sulphuric acid will immediately precipitate the glycyrrhizin from the licorice preparation, producing an unsightly mixture, and at the same time one which is nauseatingly bitter.

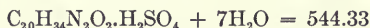
The writer knew of one physician who prescribed the sulphate

under the name of "Sulfat de pelletiere," the object of this peculiar synonym being the same as actuated the physician who prescribed the same substance under the name of "sulphatis Americanis Australis" (South American sulphate). In each case the patient had a notion that quinine was repugnant to her system, and in order to permit the administering of same without arousing her suspicions, these peculiar synonyms were employed. It must be said, however, that the first synonym is scarcely short of dangerous, inasmuch as the alkaloid pelletierine, obtained from pomegranate bark (see p. 917), is an article of commerce, and now official as the tannate, and "sulfat de pelletiere" should be left to designate the sulphate of this alkaloid.

In view of the full description of quinine and its salts just given, it is now necessary merely to insert the pharmacopœia data relative to the official salts of quinine.

Dose.—250 milligrammes (4 grains)

QUININÆ BISULPHAS. Quinine Bisulphate.



The acid sulphate [$\text{SO}_3(\text{OH})_2 \cdot \text{C}_{20}\text{H}_{34}\text{N}_2\text{O}_2 + 7\text{H}_2\text{O}$] of the alkaloid quinine. It should be kept in well-stoppered, dark amber-colored vials.

Colorless, transparent or whitish, orthorhombic crystals or small needles; odorless and having a very bitter taste. It effloresces on exposure to the air and turns yellow on exposure to the light.

Soluble in 8.5 parts of water, 18 parts of alcohol, 1770 parts of ether, 920 parts of chloroform, and in 18 parts of glycerin at 25° C. (77° F.); soluble in 0.68 part of water at 80° C. (176° F.) and in 0.5 part of alcohol at 60° C. (140° F.).

When heated the salt softens at 60° C. (140° F.), becomes semifluid at 70° C. (158° F.), and melts at about 160° C. (320° F.), with decomposition. It loses all of its water of crystallization at 100° C. (212° F.). On ignition, the salt is slowly consumed, leaving no residue.

Its aqueous solution has a strongly acid reaction and shows a blue fluorescence.

If 1 Gm. of the salt be dried at 100° C. (212° F.) until it ceases to lose weight, the residue, cooled in a desiccator, should weigh not less than 0.77 Gm. (corresponding to 7 molecules or 23 per cent. of water of crystallization).

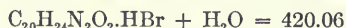
Dissolve 2 Gm. of Quinine Bisulphate, which has been dried at 50° C. (122° F.) for two hours, in 20 Cc. of distilled water, carefully neutralize with diluted sodium hydroxide T.S., and evaporate the solution to dryness on a water-bath. Complete the test by following the directions given on page 899, under *Quininæ Sulphas* (Test for Other Cinchona Alkaloids, Section II.).

Tests for Identity.—Physical tests. Given above. Thalleioquin test. See *Quinine*. Test for sulphates. See Part V.

Impurities.—Carbonizable alkaloids. See *Quinine Sulphate*.

Dose.—250 milligrammes (4 grains).

QUININÆ HYDROBROMIDUM. Quinine Hydrobromide.



[QUININÆ HYDROBROMAS, PHAR. 1890]

The hydrobromide [$\text{HBr} \cdot \text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2 + \text{H}_2\text{O}$] of the alkaloid quinine. It should be kept in well-stoppered, amber-colored vials.

White, light, silky needles; odorless and having a very bitter taste. The salt effloresces on exposure to the air.

Soluble in 40 parts of water, 0.67 part of alcohol, 16 parts of ether, 8 parts of glycerin, and very soluble in chloroform at 25° C. (77° F.); soluble in 3 parts of water at 80° C. (176° F.).

When heated to 100° C. (212° F.) the salt loses its water of crystallization. At

152° C. (305.6° F.) it begins to fuse, forming a syrupy liquid at 200° C. (392° F.). On ignition it is slowly consumed, leaving no residue.

Its solutions are neutral or slightly alkaline to litmus-paper, and when acidulated with diluted sulphuric acid shows a vivid blue fluorescence.

Ammonia water added to an aqueous solution of the salt produces a white precipitate, which is soluble in a large excess of the reagent.

If 1 Gm. of the salt be dried at 100° C. (212° F.) until it ceases to lose weight, the residue should weigh not less than 0.957 Gm. (corresponding to 1 molecule, or 4.25 per cent. of water of crystallization.)

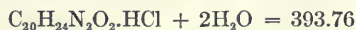
Dissolve 3 Gm. of Quinine Hydrobromide, which has been dried at 50° C. (122° F.) for two hours, in 30 Cc. of hot distilled water in an evaporating dish, and add 1.5 Gm. of crystallized sodium sulphate gradually, with constant stirring, and evaporate the liquid on a water-bath to dryness. Complete the test by following the directions given on page 899, under *Quininæ Sulphas* (Test for Other Cinchona Alkaloids, Section II.), using 30 Cc. of distilled water for maceration instead of 20 Cc., as there directed.

Tests for Identity.—Physical tests. Given above. Tests for quinine. Thalleioquin test. See Quinine. Tests for bromides. Details in Part V.

Impurities.—Carbonizable matter and morphine. See Quinine. Excess sulphate. Details in Part V. Second test for morphine. See above. Other cinchona alkaloids. See Quinine Sulphate.

Dose.—250 milligrammes (4 grains).

QUININÆ HYDROCHLORIDUM. Quinine Hydrochloride.



[QUININÆ HYDROCHLORAS, PHARM. 1890]

The hydrochloride [$\text{HCl} \cdot \text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2 + 2\text{H}_2\text{O}$] of the alkaloid quinine. It should be kept in well-stoppered, amber-colored vials.

White, silky, glistening needles; odorless and having a very bitter taste. The salt effloresces when exposed to warm air.

Soluble in 18 parts of water, 0.6 part of alcohol, 240 parts of ether, 0.8 part of chloroform, and in 8 parts of glycerin at 25° C. (77° F.); soluble in 0.4 part of water at 80° C. (176° F.).

When heated to 120° C. (248° F.), the salt loses its water of crystallization. At about 156° C. (312.8° F.) it begins to melt, but is not fully melted until the temperature reaches 190° C. (374° F.). On ignition it is slowly consumed, leaving no residue.

Its aqueous solution is neutral or faintly alkaline to red litmus-paper, and is not fluorescent except when greatly diluted, or when diluted sulphuric acid is added to it.

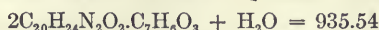
If 1 Gm. of the salt be dried at 100° C. (212° F.) until it ceases to lose weight, the residue should weigh 0.91 Gm. (corresponding to 2 molecules or 9.1 per cent. of water of crystallization.)

Dissolve 3 Gm. of Quinine Hydrochloride, which has been dried at 50° C. (122° F.) for two hours, in 30 Cc. of hot distilled water in an evaporating dish, and add 1.5 Gm. of crystallized sodium sulphate gradually, with constant stirring, and evaporate the liquid on a water-bath to dryness. Complete the test by following the directions given on page 899, under *Quininæ Sulphas* (Test for Other Cinchona Alkaloids, Section II.), using 30 Cc. of distilled water for maceration instead of 20 Cc., as there directed.

Tests for Identity.—Physical tests. Given above. Thalleioquin test. See Quinine. Test for chlorides. Details in Part V.

Impurities.—Carbonizable matter. See Quinine. Sulphates. Details in Part V. Morphine. See Quinine Hydrobromide. Other cinchona alkaloids. See Quinine Sulphate.

Dose.—250 milligrammes (4 grains).

QUININÆ SALICYLAS. Quinine Salicylate.

The salicylate $[2C_{20}H_{24}(OH)COOH \cdot C_{20}H_{24}N_2O_2 + H_2O]$ of the alkaloid quinine. It should be kept in amber-colored, well-stoppered vials.

Colorless needles, permanent in the air, but on keeping readily assuming a pinkish color.

Soluble in 77 parts of water, 11 parts of alcohol, 110 parts of ether, 37 parts of chloroform, and in 16 parts of glycerin at 25° C. (77° F.); soluble in 35 parts of water at 80° C. (176° F.) and in 11 parts of alcohol at 60° C. (140° F.).

When heated it begins to melt at 183° C. (361.4° F.), with decomposition, and at 187° C. (368.6° F.) is entirely melted to a red liquid. When ignited it is slowly consumed, without leaving a residue.

Its aqueous solution is alkaline to red litmus-paper, and has a bitter taste. When treated with diluted sulphuric acid, its aqueous solution develops a blue fluorescence.

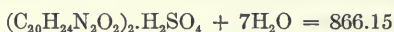
When heated at 100° C. (212° F.), to constant weight, it should lose not more than 2 per cent. in weight (absence of *excessive moisture*).

Mix 2 Gm. of Quinine Salicylate with 10 Cc. of distilled water, in a separator, add a slight excess of ammonia water, and shake the liquid with three successive portions of 25, 20, and 10 Cc. of ether, collect the ether-solution in a porcelain dish and evaporate it to dryness on a water-bath, dissolve the residue in alcohol, add just sufficient sulphuric acid to render the liquid exactly neutral, and again evaporate to dryness. Complete the test by following the directions given on page 899, under *Quininæ Sulphas* (Test for Other Cinchona Alkaloids, Section II.).

Tests for Identity.—Physical tests. Given above. *Thalleioquin* test. See Quinine. Test for salicylates. Details in Part V.

Impurities.—Excess of moisture. Details above.

Dose.—250 milligrammes (4 grains).

QUININÆ SULPHAS. Quinine Sulphate.

The sulphate $[SO_2(OH)_2 \cdot (C_{20}H_{24}N_2O_2)_2 + 7H_2O]$ of the alkaloid quinine. It should be kept in well-stoppered bottles, preferably of an amber color, and in a dark place.

White, silky, light, flexible, glistening crystals, or hard, prismatic, monoclinic needles, making a very slight and easily compressible mass, odorless, and having a persistent, very bitter taste. It effloresces rapidly when it is exposed to dry air and then becomes lustreless; when exposed to light it acquires a brownish tint. Quinine Sulphate sometimes crystallizes with 8 molecules of water of crystallization (16.18 per cent.).

Soluble in 720 parts of water, 86 parts of alcohol, 400 parts of chloroform, and in 36 parts of glycerin at 25° C. (77° F.); very difficultly soluble in ether; soluble in 45 parts of water at 80° C. (176° F.) and in 9 parts of alcohol at 60° C. (140° F.). Diluted acids increase its solubility in water, and it is easily soluble in a mixture of chloroform 2 parts and absolute alcohol 1 part.

Quinine Sulphate which has been dried over sulphuric acid melts at 205° C. (401° F.). When heated to 60° C. (140° F.), or when exposed to dry air, it loses all but 2 molecules of its water of crystallization, and upon heating it to 115° C. (239° F.) the remainder is expelled. Upon ignition the salt is slowly consumed, without leaving a residue.

Its aqueous solution is neutral to litmus-paper, and when acidulated with diluted sulphuric acid, develops a vivid blue fluorescence.

Barium chloride T.S. produces a white precipitate, insoluble in hydrochloric acid.

On treating 10 Cc. of an aqueous solution of the salt (about 1 in 1300) with 5 drops of bromine T.S., then with an excess of ammonia water, the liquid should acquire an emerald-green color (*thalleioquin*). With proper adjustment of the reagents, more diluted solutions will give a paler tint, while more concentrated ones will acquire a deeper color or throw down a green precipitate.

One Gm. of the salt should dissolve completely when heated to 50° C. (122° F.) in 7 Cc. of a mixture of 2 volumes of chloroform and 1 volume of absolute alcohol, and should remain clear on cooling (absence of *ammonium sulphate* and *inorganic salts*).

Sulphuric acid should impart to the salt not more than a faintly yellowish tint (limit of *readily carbonizable organic impurities*).

Nitric acid should not produce a red color (difference from *morphine*).

If 1 Gm. of the salt be dried at a temperature of 115° C. (239° F.), until it ceases to lose weight, the residue should weigh not less than 0.838 Gm. (indicating not more than 8 *molecules* or 16.18 *per cent. of water*).

Tests for Other Cinchona Alkaloids.—I. Dry 2 Gm. of Quinine Sulphate in a porcelain dish on a water-bath for two hours at 50° C. (122° F.).

II. Transfer the residue (which should be strictly neutral to litmus-paper) to a dry test-tube, add 20 Cc. of distilled water and place the test-tube in a water-bath; heat it for half an hour at 65° C. (149° F.) and then allow it to cool, and keep the temperature at 15° C. (59° F.) for two hours, shaking the test-tube occasionally. Filter the liquid, transfer 5 Cc. of the filtrate, having the temperature of 15° C. (59° F.), to a dry test-tube, and carefully add ammonia water (which must be of official strength and have the temperature of exactly 15° C.) until the precipitate which forms has just redissolved; not more than 6 Cc. of ammonia water should be required to produce a clear solution. If the temperature at the end of the digestion has risen to 16° C. (60.8° F.), not more than 6.5 Cc. of ammonia water should be required; if to 17° C. (62.6° F.), not more than 7 Cc. of ammonia water should be used. (In each case a clear solution indicates the absence of excessive amounts of *other cinchona alkaloids*).

Tests for Identity.—Quinine tests. Details above. Test for sulphates. Details in Part V.

Impurities.—Ammonium sulphate and other inorganic salts. Excess of moisture. Details above. Carbonizable matter and morphine. See Quinine. Other cinchona alkaloids. Details above.

Remarks.—Quinine and all its alkaloids are valuable antipyretics and antiperiodics, that is, they reduce fever and relieve malaria. The different acids combined with the alkaloid in the several salts somewhat modify the action according to the properties of the acid.

Dose.—Of quinine and its official salts, 250 milligrammes (4 grains).

Quinidine.—**Extraction.**—Obtained by concentration of the mother liquor from the recrystallization of quinine sulphate, when the impure crystals of quinidine sulphate separate out. The impure crystals of quinidine sulphate are freed from cinchonine sulphate by addition of ammonia, in which the quinidine dissolves, while the cinchonine separates. The filtered solution is treated with soda, and the quinidine thus precipitated is dissolved in diluted sulphuric acid and purified by repeated crystallizations. From the pure quinidine sulphate so made the free alkaloid is obtained by precipitation with soda.

Remarks.—Quinidine, $C_{29}H_{24}N_2O_2$, is isomeric with quinine, and, like it, is supposed to be methoxycinchonine.

It is of minor importance in medicine, having been official in the pharmacopœia of 1890 in the form of sulphate.

The salts of quinidine, like those of quinine, show a fluorescence in acid solution, and quinidine also responds to the thalleioquin test.

Cinchonidine.—**Extraction.**—Obtained from the mother liquor of quinine sulphate by precipitation with Rochelle salt, solution of this precipitate in diluted hydrochloric acid, precipitation with ammonia, and washing the precipitate free from other alkaloids with ether. The alkaloid is then redissolved and reprecipitated, and finally crystallized from alcohol.

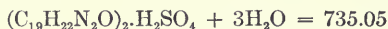
To sum up the extraction of the four important alkaloids of cinchona, the gasoline solution of these, obtained as mentioned under quinine (p. 894), is shaken out with diluted sulphuric acid. From

the concentrated acid solution the quinine and quinidine crystallize and are separated as explained under Quinidine (p. 899); the mother liquor is then treated with Rochelle salt, which precipitates the cinchonidine, while the cinchonine is obtained from the residual liquid by precipitation with soda.

Remarks.—Cinchonidine is isomeric with cinchonine, and is supposed to have the same graphic formula (p. 892). It is only sparingly soluble in ammonia water, and this explains the principle of the so-called Kerner's test for quinine, this test requiring that when an evaporated mixture of quinine and ammonium sulphate be treated with water and filtered, the filtrate should yield a clear liquid when treated with a specific quantity of water of ammonia. Rochelle salt, on the other hand, prevents precipitation of cinchonidine by ammonia, and on this fact is based the official test of distinction between cinchonidine, cinchonine, and quinidine, the two alkaloids last mentioned precipitating when so treated.

A striking test for cinchonidine is the color produced by the oxidation of the alkaloid by means of a crystal of potassium dichromate and sulphuric acid, when a brilliant, grass-green color results.

CINCHONIDINÆ SULPHAS. Cinchonidine Sulphate.



The neutral sulphate $[SO_2(OH)_2 \cdot (C_{19}H_{22}N_2O)_2 + 3H_2O]$ of an alkaloid obtained from the bark of several species of *Cinchona*.

White, glistening, silky needles or prisms, permanent in the air, odorless, having a very bitter taste. When crystallized from dilute solutions, it contains six molecules of water of crystallization, and when from concentrated solutions, three molecules.

Soluble in 63 parts of water, 72 parts of alcohol, 4400 parts of ether, and in 900 parts of chloroform at 25° C. (77° F.); soluble in 21 parts of water at 80° C. (176° F.) and in 32 parts of alcohol at 60° C. (140° F.).

When heated to 100° C. (212° F.) the salt loses its water of crystallization, and at 203° C. (397.4° F.) it darkens, and then melts at 205.3° C. (401.5° F.). At a higher temperature it ignites and is consumed without leaving a residue.

The aqueous solution is neutral or only faintly alkaline to phenolphthalein T.S. or litmus T.S., and is levogyrate.

On adding ammonia water to the aqueous solution of the salt, a white precipitate (cinchonidine) is produced, which is but slightly soluble in ammonia, but which, when freshly precipitated, dissolves in 10 parts of ether, the greater part afterward separating in crystals.

If sulphuric acid be added to a small quantity of the salt, not more than a faintly yellowish color should be developed (limit of *readily carbonizable organic impurities*). Upon adding to this liquid a crystal of potassium dichromate, a yellowish-green color is produced, which gradually changes to grass-green.

If 1 Gm. of the salt be dried at 100° C. (212° F.) until it ceases to lose weight, the residue, cooled in a desiccator, should weigh not less than 0.920 Gm. (absence of an *undue amount of water*).

A solution of the salt (1 in 1000) in diluted sulphuric acid should not exhibit more than a faint blue fluorescence (absence of more than traces of *quinine* or *quinidine sulphates*).

If 0.5 Gm. of the salt be macerated, with frequent agitation, at the ordinary temperature, with 20 Cc. of water, 0.5 Gm. of potassium and sodium tartrate then added, the maceration continued, under repeated agitation, for one hour at 15° C. (59° F.), and the mixture filtered, the addition of 1 drop of ammonia water to the filtrate should not produce more than a slight turbidity (absence of more than small quantities of *cinchonine* or *quinidine sulphates*).

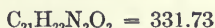
Tests for Identity.—For cinchonidine. Given above. For sulphates. Details in Part V.

Impurities.—Carbonizable matter, excess of water, other cinchona alkaloids. Details above.

Remarks.—This alkaloidal salt enjoyed a large sale some twenty-five years since, when the high price of quinine sulphate (which then sold at \$5 an ounce) made that important alkaloid a luxury. It is used like quinine, but is not so effective.

Dose.—250 milligrammes (4 grains).

STRYCHNINA. Strychnine.



An alkaloid obtained from *Nux Vomica*, and also obtainable from other plants of the *Loganiaceae*.

Colorless, transparent, prismatic crystals, or a white crystalline powder; odorless and having an intensely bitter taste, perceptible even in solutions of 1 in 700,000. *Strychnine should be tasted with extreme caution.* Permanent in the air.

Soluble in 6400 parts of water, 110 parts of alcohol, 5500 parts of ether, 6 parts of chloroform, 150 parts of benzene, and in 180 parts of amyl alcohol at 25° C. (77° F.); soluble in 3000 parts of water at 80° C. (176° F.) and in 28 parts of alcohol at 60° C. (140° F.).

Its melting-point is 268° C. (514.4° F.). Upon ignition it is consumed, leaving no residue.

Its solutions are lævogyrate and alkaline to litmus-paper.

Sulphuric acid containing 1 per cent. of ammonium vanadate produces with Strychnine a deep violet-blue color, changing to a deep purple, and finally to a cherry-red. Sulphuric acid containing a trace of potassium iodate produces a violet color, changing momentarily to reddish-purple.

If 0.1 Gm. of Strychnine dissolved in a few drops of nitric acid be evaporated to dryness, and a few drops of ammonia water added to the yellow residue, an orange color will be produced, which will turn reddish-purple, and finally brown, on the addition of a small amount of alcoholic potassium hydroxide T.S.

Sulphuric acid should produce no color (absence of *sugar* and *other readily carbonizable organic impurities*), but on adding a fragment of potassium dichromate, a deep blue color is momentarily produced, changing to deep violet, then to purplish-red, cherry-red, and finally to orange or yellow.

Nitric acid, when added to a crystal of Strychnine on a white porcelain surface, should not produce more than a faintly pink color (limit of *brucine*).

Tests for Identity.—Physical tests. See above.

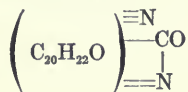
Impurities.—Carbonizable substances. See Atropine (p. 884). Brucine. Details above.

Extraction.—*Nux vomica* is extracted with very dilute acid, and the alkaloids precipitated from the acid liquid by use of milk of lime. The precipitate is freed from mucilage, etc., by washing with water, and from brucine by use of cold diluted alcohol.

It is then treated with boiling alcohol, which dissolves the strychnine, and this is decolorized by treatment with animal charcoal. The alkaloid is eventually precipitated from the alcoholic solution by treatment with alkali.

Remarks.—This alkaloid was discovered by Pelletier and Caventou in 1818. It is the active principle of *nux vomica* and *Ignatia bean*, from which it is extracted as explained above.

The structural composition of strychnine is still a matter of doubt, the only points established being shown in the graphic formula:



The synthesis of strychnine has not yet been accomplished.

Interesting among the reactions of strychnine is its behavior under different oxidizing agents. Thus, when strychnine is first dissolved in concentrated sulphuric acid, which it does without any change in color, and if a small crystal of potassium dichromate is added, a violet color changing to tints of red, then orange, and yellow results. This test is said to detect one part of strychnine in 500,000. It is, however, open to fallacy, Professor J. U. Lloyd having shown that a mixture of morphine and hydrastine will give a similar color reaction. Professor Lloyd makes the confusion of such a combination for strychnine a dramatic feature of one of his romances.

A very prominent characteristic of strychnine is its intensely bitter taste, the bitterness being shown in a dilution of 1 to 700,000, which can be stated more strikingly by saying that this dilution represents about a grain of strychnine to twelve gallons of water.

Strychnine is a powerful nervous stimulant, and is usually dispensed as the sulphate.

Dose.—1 milligramme ($\frac{1}{84}$ grain).

It is recognized by the pharmacopœia in the form of the free alkaloid, and also as the nitrate and the sulphate.

STRYCHNINÆ NITRAS. Strychnine Nitrate.



The nitrate $[\text{NO}_3\text{OH} \cdot \text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2]$ of the alkaloid Strychnine. It should be kept in well-stoppered vials.

Colorless, glistening needles; odorless and having an intensely bitter taste. *Strychnine Nitrate should be tasted with extreme caution.* Permanent in the air.

Soluble in 42 parts of water, 120 parts of alcohol, 156 parts of chloroform, and in 60 parts of glycerin at 25° C. (77° F.); soluble in 8 parts of water at 80° C. (176° F.) and in 60 parts of alcohol at 60° C. (140° F.); insoluble in ether.

When heated the salt decomposes, but it does not melt, and when ignited it is consumed without leaving a residue.

Its aqueous solutions are neutral to litmus-paper and lævogyrate.

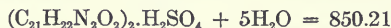
The addition of sulphuric acid to a crystal of Strychnine Nitrate on a white porcelain surface should not produce more than a faintly yellow color (limit of *brucine*).

Tests for Identity.—Physical tests. Given above. Color tests. See Strychnine. Tests for nitrate. Details in Part V.

Impurity.—Brucine. Details above.

Dose.—1 milligramme ($\frac{1}{84}$ grain).

STRYCHNINÆ SULPHAS. Strychnine Sulphate.



The sulphate $[\text{SO}_2(\text{OH})_2 \cdot (\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2)_2 + 5\text{H}_2\text{O}]$ of the alkaloid strychnine. It should be kept in well-stoppered vials.

Colorless or white prismatic crystals, or a white, crystalline powder; odorless and having an intensely bitter taste, perceptible even in solutions of 1 in 700,000. *Strychnine Sulphate should be tasted with extreme caution.* Efflorescent in dry air.

Soluble in 31 parts of water, 65 parts of alcohol, and in 325 parts of chloroform at 25° C. (77° F.); soluble in 6 parts of water at 80° C. (176° F.) and in 20 parts of alcohol at 60° C. (140° F.); insoluble in ether.

When heated at 100° C. (212° F.) the salt loses its water of crystallization (10.59 per cent.), and when anhydrous melts at 200° C. (392° F.). Upon ignition it is consumed, leaving no residue.

Nitric acid, when added to a crystal of Strychnine Sulphate on a white porcelain surface, should not produce more than a faintly yellow color (limit of *brucine*).

Tests for Identity.—Physical tests. Given above. Color tests. See Strychnine. Test for sulphates. Details in Part V.

Impurity.—Brucine. Details above.

Remarks.—Both the sulphate and the nitrate are used like the alkaloid, strychnine, and in the same dose.

Dose.—1 milligramme ($\frac{1}{84}$ grain).

Brucine is an unofficial alkaloid, associated with strychnine in *nux vomica* and *ignatia*. It is of little importance in pharmacy and medicine, except that some commercial strychnine has not been fully purified from brucine. This is detected by the fact that brucine when treated with concentrated nitric acid gives a brilliant red tint, while strychnine will dissolve practically colorless.

VERATRINA. Veratrine.

A mixture of alkaloids obtained from the seed of *Asagraea officinalis* (Chamisso and Schlechtendal) Lindley (Fam. *Liliaceæ*). It should be kept in well-stoppered, amber-colored vials.

A white, or grayish-white, amorphous powder, odorless, but causing intense irritation and sneezing when even a minute quantity reaches the nasal mucous membrane; having an acrid taste and leaving a sensation of tingling and numbness on the tongue. *It should be tasted with great caution.* Slightly hygroscopic in moist air.

Soluble in 1750 parts of water, 2.2 parts of alcohol, 3 parts of ether, and in 1 part of chloroform at 25° C. (77° F.); soluble in 1300 parts of water at 80° C. (176° F.); very soluble in benzene and amyl alcohol; insoluble in petroleum benzin.

When heated to 145° C. (293° F.) it softens, and melts at 152° C. (305.6° F.). Upon ignition it is consumed, leaving no residue.

Its alcoholic solutions are alkaline to red litmus-paper.

On triturating Veratrine with sulphuric acid, in a glass mortar, the yellow or orange-red solution exhibits, by reflected light, a greenish fluorescence, which becomes more intense upon the addition of an equal volume of acid. Upon standing, the solution gradually assumes a deep red color.

Sulphuric acid when heated with Veratrine gives a cherry-red color. Sulphuric acid containing a trace of selenous acid produces a brownish-green color. Sulphuric acid added to a mixture of 1 part of Veratrine and 6 parts of sugar produces a green color, changing to blue, and the mixture then becomes colorless.

Tests for Identity.—Given above.

Extraction.—Powdered cevadilla seeds are extracted with 90 per cent. alcohol, the percolate concentrated and poured into a large quantity of water, thus precipitating resin, fat, etc. The aqueous liquid is then made alkaline with ammonia, and the precipitated veratrine purified by washing with water, dissolving in diluted hydrochloric acid, and precipitating with ammonia.

Remarks.—Veratrine is a mixture of alkaloids obtained from cevadilla seeds. The word veratrine is unfortunate, inasmuch as it would suggest that the alkaloid is obtained from the official veratrum. This is not the case, the alkaloid having originally been discovered in cevadilla seeds in 1818, by Meissner, who called the alkaloid *sabadiline*, suggesting, however, that if the same alkaloid was found in other species of veratrum, it might be well to call it veratrine. The same year, however, Pelletier and Caventou isolated veratrine from white veratrum, and from this fact the alkaloid obtained its name.

The chemistry of this alkaloid has been comparatively little

studied. As above mentioned, it is now considered a mixture of alkaloids. It is a quinoline derivative, as is shown by its decomposition products.

Its chief reactions, as cited above, are the colors produced with concentrated sulphuric acid, the tint ranging from yellow to cherry red, according to conditions of mixing.

It will be noted in the alkaloidal reactions of the new pharmacopœia that selenous acid plus sulphuric acid is given a place as an alkaloidal color reagent—in the case of veratrine a green-brown color is produced.

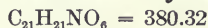
Veratrine is used internally very seldom, and then as a sedative. Its chief use is externally as a destroyer of vermin and in some forms of skin trouble, in which case it is usually dispensed as the official *oleate of veratrine* or as the ointment.

Dose.—2 milligrammes ($\frac{1}{30}$ grain).

GROUP IV.—THE ISOQUINOLINE DERIVATIVES

Isoquinoline is a substance having the same empiric formula as quinoline, the only difference between the two being in the location of the nitrogen atom, which can best be shown by the graphic formula given on p. 905.

HYDRASTINA. Hydrastine.



An alkaloid obtained from Hydrastis. It should be kept in well-stoppered bottles.

White to creamy white, glistening prisms, sometimes of large size, possessing a bitter taste, and permanent in the air. It contains no water of crystallization.

Almost insoluble in water at 25° C. (77° F.); soluble in 135 parts of alcohol, 124 parts of ether, and in 2 parts of chloroform at 25° C. (77° F.); soluble in 4000 parts of water at 80° C. (176° F.) and in 17 parts of alcohol at 60° C. (140° F.); easily soluble in benzene.

Hydrastine melts at 131° C. (267.8° F.).

It shows an alkaline reaction to moistened litmus-paper and is lævogyrate.

Sulphuric acid produces a yellow color when added to Hydrastine, and on heating a purple color is developed.

Sulphuric acid containing a trace of molybdic acid gives a green color, changing to olive-green and then to brown; nitric acid yields a reddish-yellow color; sulphuric acid containing a trace of selenous acid gives a yellowish-red color, changing to brown.

Sulphuric acid containing a trace of potassium dichromate produces a red color, changing to brown.

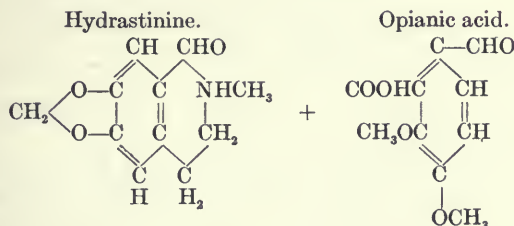
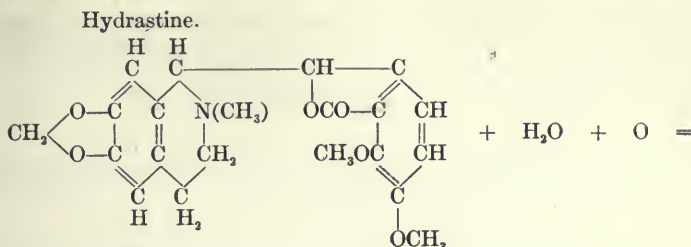
If a crystal of Hydrastine be dissolved in diluted sulphuric acid and a solution of potassium permanganate (1 in 10) be added, a blue fluorescence will be developed (distinction from hydrastinine).

Tests for Identity.—Given above.

Impurities.—Hydrastinine. Details above.

Extraction.—A decoction of hydrastis is made with water containing acetic acid. This is evaporated to a syrup and mixed with three volumes of 20 per cent. sulphuric acid. This causes the separation of the crystals of bisulphate of berberine (yellow hydrastine). The mother liquor is made alkaline with ammonia, and the crude precipitated hydrastine is purified by recrystallization with alcohol or acetic acid.

Hydrastine, on treatment with oxidizing agents (diluted nitric acid), splits into hydrastinine and opianic acid:

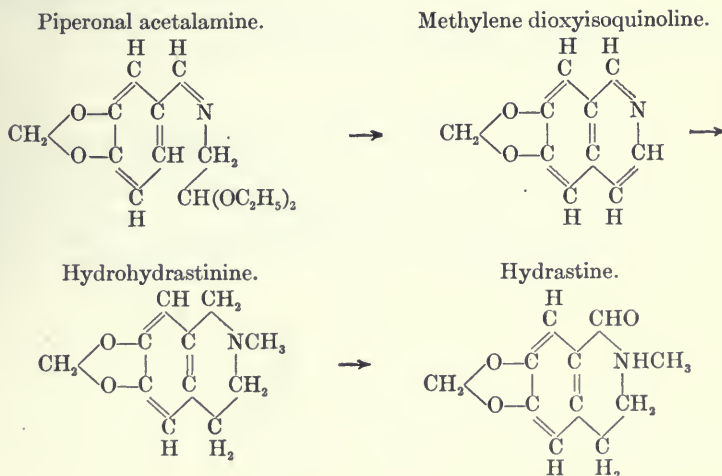


Hydrastine occurs in handsome white crystals, soluble in 135 parts of alcohol and insoluble in water. It is largely used as an astringent, and especially in gonorrheal washes. Unfortunately, two articles are known in commerce as hydrastine, the white crystals above described, which are quite expensive, and the so-called *yellow hydrastine*, which is nothing less than berberine.

Dose.—10 milligrammes ($\frac{1}{4}$ grain).

Berberine is an unofficial alkaloid obtained from hydrastis and also from the root of *Berberis vulgaris*; hence its name. To all these drugs it transmits its characteristic yellow color. Its structural formula is similar to hydrastine, although even more complex.

Hydrastinine.—This is an artificial alkaloid obtained from hydrastine by treatment with appropriate oxidizing agents. The synthesis of hydrastinine from piperonal acetalamine has been accomplished, the successive stages of this synthesis being graphically shown below:



The graphic formula for hydrastinine just given, which adds up to $C_{11}H_{13}NO_3$, is the one proposed by Eykman, while that given by the pharmacopœia under hydrastinine hydrochloride ($C_{11}H_{11}NO_2$) is the one reported by Freund. It will be seen that the difference between hydrohydrastinine and hydrastinine is that, in the latter, the nitrogen ring of the isoquinoline structure is broken.

HYDRASTININÆ HYDROCHLORIDUM. Hydrastinine Hydrochloride.



[HYDRASTININÆ HYDROCHLORAS, PHARM. 1890]

The hydrochloride [$HCl \cdot C_{11}H_{11}NO_2$] of an artificial alkaloid derived from hydrastine. It should be kept in well-stoppered bottles.

Light yellowish needles, or a yellowish-white, crystalline powder; odorless and having a bitter taste.

Very soluble in cold and hot water and in alcohol; soluble in 286 parts of chloroform and 1300 parts of ether at $25^\circ C.$ ($77^\circ F.$).

Hydrastinine Hydrochloride melts at $212^\circ C.$ ($413.6^\circ F.$).

Its aqueous solution, especially when highly diluted, shows a blue fluorescence and is neutral to litmus-paper. On ignition the salt should be completely consumed.

In an aqueous solution of the salt (1 in 20), bromine T.S. produces a yellow precipitate, which should be perfectly soluble in ammonia water, forming an almost colorless solution.

In an aqueous solution of the salt, potassium dichromate T.S. produces a precipitate which redissolves if gently heated, but on cooling the solution it separates in glistening needles.

If ammonia water be added to an aqueous solution of Hydrastinine Hydrochloride (1 in 20), no turbidity should be produced.

On slowly adding to a solution of 0.2 Gm. of the salt in 3 Cc. of water 4 to 5 drops of a solution of sodium hydroxide (1 in 7), each drop will cause a milky turbidity, which disappears again on shaking. From this solution, after standing for some time, pure white hydrastinine should separate, and the supernatant fluid should be almost colorless.

With a crystal of the salt, sulphuric acid or nitric acid produces a deep yellow color; sulphuric acid with a trace of nitric acid, a reddish-brown color; sulphuric acid with a crystal of ammonium vanadate, a light brown color changing to dark brown.

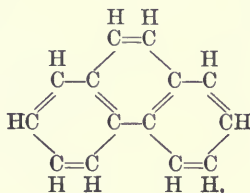
Tests for Identity.—Given above.

Remarks.—It is worth noting that an aqueous solution of this salt gives a blue fluorescence, somewhat similar to that exhibited by quinine. It is used as a uterine astringent.

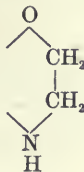
Dose.—30 milligrammes ($\frac{1}{2}$ grain).

GROUP V.—PHENANTHRENE DERIVATIVES

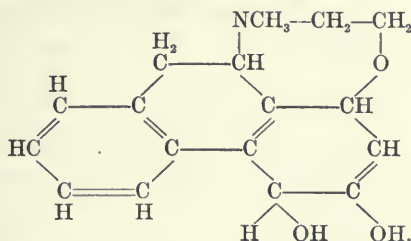
The difficulty of making a clear-cut definition for alkaloids is shown in the case of morphine and its allied opium alkaloids. The definition for alkaloids given on p. 875 states that they are derivatives of pyridine, quinoline, or allied bodies, and yet latest investigations seem to show that morphine, one of the most prominent alkaloids, is not a derivative of isoquinoline as long supposed, but is derived from phenanthrene, an isomer of anthracene, and, therefore, containing no nitrogen. The graphic formula of phenanthrene is—



and the nitrogen of morphine is supposed to be in a characteristic grouping known as the *morpholine ring*:

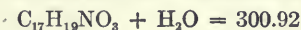


The graphic formula of morphine is supposed to be—



The synthesis of morphine has not yet been accomplished.

MORPHINA. Morphine.



An alkaloid obtained from Opium.

Colorless or white, shining, rhombic prisms, or fine needles, or a crystalline powder, odorless and having a bitter taste; permanent in the air. It loses all of its water of crystallization at 100° C. (212° F.).

Soluble in 3330 parts of water, 100 parts of lime water, 168 parts of alcohol, 4464 parts of ether, 1800 parts of chloroform, 113.5 parts of amyl alcohol, and in 525 parts of acetic ether at 25° C. (77° F.); soluble in 1040 parts of water at 80° C. (176° F.) and in 76 parts of alcohol at 60° C. (140° F.); insoluble in benzene.

When heated to about 200° C. (392° F.) it assumes a brown color, and then melts at 254° C. (489.2° F.). Upon ignition it is slowly consumed without leaving a residue.

Its aqueous solution shows an alkaline reaction to red litmus-paper.

Sulphuric acid (free from nitrous compounds), added to Morphine, produces no color or only a slight yellowish tint, but on heating a brown color is developed.

Sulphuric acid containing a crystal of potassium iodate gives with Morphine a dark brown color. (*Codeine* yields a moss-green color, changing to brown, and *narcotine* a cherry-red color.)

Sulphuric acid containing a trace of selenous acid gives with Morphine a blue color, changing to green and then to brown. (*Codein* yields a green color, changing to blue, and afterward to grass-green; *narcotine* gives a green color, changing to brown, and then to cherry-red.)

Nitric acid produces with Morphine an orange-red color, fading to yellow (difference from *quinine*). Sulphuric acid containing a trace of molybdic acid gives a purple color, changing to blue. Sulphuric acid, containing in each Cc. one drop of solution of formaldehyde, yields an intense purple color.

Mercuric potassium iodide T.S. produces in a solution of Morphine a white gelatinous precipitate.

A solution of sodium phosphomolybdate (1 in 20) produces in solutions of Morphine a yellow precipitate soluble in ammonia water.

Sulphuric acid containing a crystal potassium dichromate gives no color at first, but after a time a green color (absence of *strychnine*, which yields a purple color, or of *acetanilide*, which gives a crimson color, changing to green).

Morphine solutions, when heated with an aqueous solution of potassium ferricyanide containing a drop of neutral ferric chloride solution, give a deep blue solution, from which, after standing, a blue precipitate separates (difference from *codeine*).

If 0.1 Gm. of Morphine be dissolved in 10 Cc. of diluted hydrochloric acids, no red coloration should be produced by the addition of a few drops of ferric chloride T.S. (absence of *meconic acid* or *meconates*).

If to a neutral solution of Morphine (1 in 100), made by the careful addition of diluted sulphuric acid, a few drops of ferric chloride T.S. be added, a blue color will be produced, which is destroyed by acids, alcohol, or by heating.

On adding 4 Cc. of potassium hydroxide T.S. to 0.2 Gm. of Morphine, a clear, colorless solution, free from any undissolved residue, should result (absence of, and difference from, various *other alkaloids*), and no odor of ammonia should be noticeable (absence of *ammonium salts*).

Tests for Identity.—Given above.

Impurities.—Codeine, narcotine, quinine, strychnine, acetanilide, meconic acid, other alkaloids, and ammonium salts. Details above.

Extraction.—An infusion of opium is concentrated, and then made alkaline with sodium carbonate. The precipitate is washed with water to remove excess of alkali; with alcohol, to remove lesser alkaloids, color, etc.; and then treated with diluted acetic acid, which dissolves the morphine and leaves the narcotine. The morphine is then precipitated from the acid solution by addition of ammonia water.

Remarks.—Morphine is, with the possible exception of quinine, the most important of the alkaloids. It is also interesting as being the first alkaloid to be isolated, its separation from opium being accomplished by Sertürner, an apothecary of Eimbeck, in 1804.

Important points in the tests just given are: The color produced by the alkaloid in combination with sulphuric acid and potassium iodate and the blue-green-brown color-play produced by action of morphine on sulphuric acid and selenous acid. These tests distinguish morphine from the other opium alkaloids.

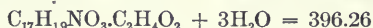
Morphine, when sprinkled upon concentrated nitric acid, gives a red color similar to brucine. The reaction, however, differs from the brucine reaction by being less permanent, and is of value, distinguishing morphine from quinine.

Morphine differs from most other alkaloids by being soluble in potassa and soda, other alkaloids being precipitated from solution by addition of alkalis. It is used like opium as a hypnotic and sedative.

Dose.—10 milligrammes ($\frac{1}{8}$ grain).

Morphine is official in the form of the free alkaloid, also as acetate, hydrochloride, and the sulphate.

MORPHINÆ ACETAS. Morphine Acetate.



The acetate [$\text{CH}_3\text{COOH} \cdot \text{C}_{17}\text{H}_{19}\text{NO}_3 + 3\text{H}_2\text{O}$] of the alkaloid morphine should be kept in well-stoppered, dark amber-colored vials; a minute quantity of free acetic acid should be present to prevent decomposition.

A white or yellowish-white, crystalline or amorphous powder, having a faintly acetous odor and a bitter taste. It gradually loses acetic acid when exposed to air and becomes less soluble in water.

Soluble in 2.25 parts of water, 21.6 parts of alcohol, 480 parts of chloroform, and in 5.2 parts of glycerin at 25° C. (77° F.); soluble in 2 parts of water at 80° C. (176° F.) and in 2.5 parts of alcohol at 60° C. (140° F.); insoluble in ether.

When heated, the salt loses water and acetic acid, and melts at about 200° C. (392° F.). When ignited, it is consumed completely, leaving no residue.

The addition of diluted ammonia water in slight excess to an aqueous solution of Morphine Acetate causes a white precipitate, which, when collected and washed, should conform to the reactions and tests given under *Morphina*.

On adding sulphuric acid to the salt, vapors of acetic acid are evolved.

Ferric chloride T.S. produces, in neutral aqueous solutions of the salt, a blue color, destroyed by acids, alcohol, or by heating.

The color tests for the identification of Morphine Acetate and those showing the absence of impurities are identical with those described under *Morphina*.

Tests for Identity.—Given above.

Dose.—15 milligrammes ($\frac{1}{4}$ grain).

MORPHINÆ HYDROCHLORIDUM. Morphine Hydrochloride.



[MORPHINÆ HYDROCHLORAS, PHARM. 1890]

The hydrochloride [$\text{HCl} \cdot \text{C}_{17}\text{H}_{19}\text{NO}_3 + 3\text{H}_2\text{O}$] of the alkaloid morphine should be kept in well-stoppered, amber-colored vials.

White, silky, glistening needles or microcrystalline cubes, or a white, crystalline powder, odorless, and having a bitter taste; permanent in the air. It loses its water of crystallization at 100° C. (212° F.).

Soluble in 17.2 parts of water and in 42 parts of alcohol at 25° C. (77° F.); soluble in 0.5 part of water at 80° C. (176° F.) and in 35.5 parts of alcohol at 60° C. (140° F.); insoluble in ether and in chloroform.

When heated to 250° C. (482° F.) it assumes a brown color, and on higher heating, chars without melting. On ignition, it is slowly consumed, leaving no residue.

Its aqueous solution is neutral to litmus-paper.

On shaking an aqueous solution of the salt with diluted ammonia water in slight excess, a crystalline precipitate is formed, which, when collected and washed, should respond to all tests and reactions given under *Morphina*.

Silver nitrate T.S. produces a white precipitate, insoluble in nitric acid.

Ferric chloride T.S. produces, in neutral aqueous solutions of the salt, a blue color, which is destroyed by acids, alcohol, or by heating.

On adding potassium carbonate T.S. to a solution of the salt (1 in 30), a white precipitate should be formed, which should dissolve in chloroform without color (absence of *apomorphine*).

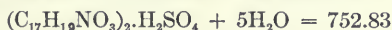
The color tests for the identification of Morphine Hydrochloride and those showing the absence of impurities are identical with those described under *Morphina*.

Tests for Identity.—Given above.

Impurity.—Apomorphine. Details above.

Dose.—15 milligrammes ($\frac{1}{4}$ grain).

MORPHINÆ SULPHAS. Morphine Sulphate.



The sulphate [$\text{SO}_4(\text{OH})_2 \cdot (\text{C}_{17}\text{H}_{19}\text{NO}_3)_2 + 5\text{H}_2\text{O}$] of the alkaloid morphine should be kept in well-stoppered, amber-colored vials.

White, feathery, acicular, silky crystals, or in cubical masses, odorless, permanent in the air, and having a bitter taste. It loses three molecules of water of crystallization at 100° C. (212° F.) and the remaining two at 130° C. (266° F.).

Soluble in 15.3 parts of water and in 465 parts of alcohol at 25° C. (77° F.); soluble in 0.6 part of water at 80° C. (176° F.) and in 187 parts of alcohol at 60° C. (140° F.); insoluble in ether and chloroform.

When heated to about 250° C. (482° F.) the salt assumes a brown color, and then chars without melting. When ignited, it is very slowly consumed, leaving no residue.

Its aqueous solution is neutral to litmus-paper.

On adding a dilute solution of ammonia water in slight excess to its aqueous solution, and vigorously shaking the liquid, a precipitate is formed which, when collected and washed, should respond to all the tests and reactions given under *Morphina*.

Barium chloride T.S. produces a white precipitate, insoluble in hydrochloric acid.

Ferric chloride T.S., when added to neutral aqueous solutions of the salt, produces a blue color, which is destroyed by acids, alcohol, or by heating.

The color tests for the identification of Morphine Sulphate, and those showing the absence of impurities, are identical with those described under *Morphina*.

Tests for Identity.—Given above.

Remarks.—All three of these morphine salts are used like the alkaloid. Of them, the sulphate is the most largely used.

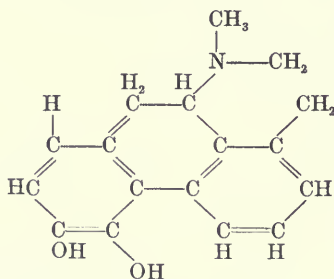
Magendie's solution is an aqueous solution of morphine sulphate, 16 grains to the fluidounce. *Syrup of Morphine*, N. F., 1 grain morphine to the fluidounce, is used in the south, where a mixture of equal quantities of the syrups of squill, tolu, morphine, and ipecac is prescribed as "*four syrups*," or "*syrupus quatuor*."

Dose.—Of all the morphine salts, 15 milligrammes ($\frac{1}{4}$ grain).

Heroine.—This is a diacetic acid ester of morphine, prepared from morphine by the replacing of its two hydroxyl groups by acetyl groups. It is supposed to be preferable to morphine because devoid of evil after-effects, but how much of this is due to the judicious advertising of the manufacturer remains to be seen. In fact, although in use for only a few years, we have already heard of heroine fiends. It is given as a hypnotic as a substitute for morphine in doses of from 5 to 10 milligrammes.

Apomorphine.—This interesting artificial alkaloid is prepared by heating morphine with concentrated hydrochloric acid in sealed tubes to 150° C. The sealed tubes in this case are tubes of glass about a quarter of an inch thick and three feet long, originally in the shape of an ordinary test-tube. The ingredients are placed in this tube, and then the upper portion is drawn to a fine point by heating in a blast lamp flame. In this way the ingredients are sealed within the walls of glass. This tube is then wrapped up in paper and placed within an iron tube, stopped at one end with an appropriate cap. This tube is then placed in a suitable oven and is heated for the requisite time, and after cooling for a time the sealed tip is opened cautiously to permit the escape of the compressed gases, and is finally broken by cutting with a glass file.

The formula for apomorphine differs from that of morphine simply by the loss of one molecule of water, which is removed by the action of concentrated hydrochloric acid, as mentioned above. It is supposed to be:



APOMORPHINÆ HYDROCHLORIDUM. Apomorphine Hydrochloride.

$$\text{C}_{17}\text{H}_{17}\text{NO}_2 \cdot \text{HCl} = 301.34$$

[APOMORPHINÆ HYDROCHLORAS, PHARM. 1890]

The hydrochloride [$\text{HCl} \cdot \text{C}_{17}\text{H}_{17}\text{NO}_2$] of an alkaloid prepared from morphine by the abstraction of one molecule of water. It should be kept in small, dark amber-colored vials, which have been previously rinsed with diluted hydrochloric acid and dried.

Minute grayish-white monoclinic prisms, glistening, odorless, having a slightly bitter taste and acquiring a greenish tint upon exposure to light and air.

Soluble in 39.5 parts of water, 38.2 parts of alcohol, 1864 parts of ether, and in 3800 parts of chloroform at 25° C. (77° F.); soluble in 16 parts of water at 80° C. (176° F.) and in 30 parts of alcohol at 60° C. (140° F.).

The salt decomposes between 200° and 210° C. (392° and 410° F.) and melts at 263° C. (507° F.).

The aqueous solution shows a neutral reaction with litmus-paper.

If the salt imparts at once an emerald-green color to 100 parts of water on being shaken with it a few times in a test-tube, it should be rejected.

Apomorphine Hydrochloride is not colored when treated with sulphuric acid; with nitric acid a deep purple color fading to orange is produced; with sulphuric acid containing a trace of selenous acid, a dark blue color, fading to violet, and then turning black; with sulphuric acid containing a trace of nitric acid, a blood-red color, fading to orange; with sulphuric acid containing a trace of ferric chloride, a pale blue color; with sulphuric acid containing a trace of ammonium vanadate, a violet-blue color, changing to deep greenish-blue; with sulphuric acid containing a little paraldehyde, a green color, fading to reddish-brown; with sulphuric acid containing potassium iodate, a black color, changing to brown, and finally to pale brown.

If sulphuric acid be added to a crystal of Apomorphine Hydrochloride and a crystal of potassium nitrate, the latter is colored red, and on stirring with a glass rod the solution becomes green, then blue, then purple, and finally cherry-red. Acetic acid dissolves the salt without color, but on adding a trace of potassium iodate, the solution turns blood-red, changes to purple, and on adding a little ether and shaking, the latter assumes a blue color.

Gold chloride T.S. produces a reddish-purple precipitate in a solution of the salt. Diluted ferric chloride T.S. colors Apomorphine Hydrochloride solution red (distinction from *morphine*, which, by the same test, is colored blue).

Silver nitrate T.S. added to the aqueous solution of the salt throws down a white precipitate, insoluble in nitric acid, soon turning black by reduction to metallic silver, and instantly reduced by the addition of ammonia water.

If 0.05 Gm. of the salt be shaken with a solution made by dissolving 0.05 Gm. of ferrous sulphate in 10 Cc. of water, the solution will gradually turn blue and then black; upon the addition of some alcohol the solution resumes its blue color (difference from *codeine*, *morphine*, *narceine*, and *narcotine*).

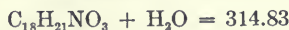
Tests for Identity.—Given above.

Impurities.—*Morphine*, *codeine*, *narceine*, and *narcotine*. Details above.

Remarks.—Apomorphine hydrochloride dissolves in water in a colorless solution, but on standing it changes to a greenish tint. The nature of the decomposition is not yet fully understood, but is worth bearing in mind in prescription practice. This alkaloid salt is emetic, being of particular value employed hypodermically in cases of attempted suicide by poisoning.

Dose.—Expectorant, 2 milligrammes ($\frac{1}{80}$ grain); emetic, 5 milligrammes ($\frac{1}{16}$ grain).

CODEINA. Codeine.



An alkaloid $[\text{C}_{17}\text{H}_{18}(\text{CH}_3)\text{NO}_3 + \text{H}_2\text{O}]$ obtained from Opium, or prepared from morphine by methylation. It should be kept in well-stoppered, amber-colored vials.

White or nearly translucent, orthorhombic prisms, octahedral crystals, or a crystalline powder; odorless and having a faintly bitter taste; slightly efflorescent in warm air.

Soluble in 88 parts of water, 1.6 parts of alcohol, 12.5 parts of ether, and 0.66 part of chloroform at 25° C. (77° F.); soluble in 59 parts of water at 80° C. (176° F.) and in 0.92 part of alcohol at 60° C. (140° F.).

At 100° C. (212° F.) it loses its water of crystallization and melts at 154.9° C. (310.8° F.). When heated in an insufficient amount of water for complete solution, it melts to oily drops, which crystallize on cooling. It leaves no residue on incineration.

Its aqueous solution is alkaline to litmus-paper and is lœvogyrate.

Sulphuric acid (free from nitrous compounds) produces either no color or a slight pinkish tint, which disappears within two minutes, but on heating a violet color is developed. (The presence of nitrous compounds causes a pink color in the cold.)

Sulphuric acid containing a trace of ferric chloride produces with Codeine a violet-blue color; sulphuric acid heated with a drop of nitric acid added gives a blood-red color; sulphuric acid containing a trace of selenous acid produces a green color, changing rapidly to blue, and then slowly back to grass-green (with morphine it gives a blue color, changing to green, and then to brown); sulphuric acid and Codeine develop no color, but on adding a drop of solution of formaldehyde a violet-blue color is produced (morphine giving an intense purple color).

On dissolving a small crystal of potassium ferricyanide in 10 Cc. of water, adding 1 drop of ferric chloride T.S., and then 1 Cc. of Codeine solution (1 in 100), no blue color should be produced at once (absence of *morphine*).

If 1 drop of a diluted nitric acid solution (1 drop in 200 Cc. of water) be added to a solution 0.03 Gm. of Codeine in 2 Cc. of sulphuric acid, a bluish-red tint, gradually changing to blue, will be developed.

On sprinkling 0.05 Gm. of Codeine upon 2 Cc. of nitric acid (sp. gr. 1.200) the crystals will turn red, but the acid will acquire only a yellow color (difference from, and absence of, *morphine*).

Tests for Identity.—Given above.

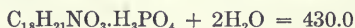
Impurity.—Morphine. Details above.

Extraction.—The aqueous infusion of opium is concentrated and treated with calcium chloride, which holds the alkaloids in solution as hydrochlorides, while the meconic acid, color, etc., are precipitated. The filtrate is concentrated, when the hydrochlorides of codeine and morphine crystallize. The mixed salts are redissolved in water and the solution made alkaline with ammonia, which precipitates the morphine. The filtrate on concentration yields crystals of codeine hydrochloride, and the free alkaloid is obtained from this by treatment with potassa. The above process is also another of the many methods for obtaining morphine from opium.

Remarks.—Codeine is another of the numerous alkaloids found in opium, and can be obtained synthetically by treatment of morphine with potassium hydrate and methyl iodide. This synthesis and other investigations show that it is methyl ether of morphine.

Characteristic reactions of codeine are, as of many alkaloids already studied, the behavior of the substance with concentrated sulphuric acid and various oxidizing or reducing agents, such as nitric acid, ferric chloride, selenous acid, and formaldehyde. These tests are of value in distinguishing codeine from morphine, as is also the fact that codeine turns yellow with nitric acid and *not* red, as does morphine.

CODEINÆ PHOSPHAS. Codeine Phosphate.



The phosphate $[\text{PO}(\text{OH})_2(\text{C}_{17}\text{H}_{18}(\text{CH}_3)\text{NO}_3) + 2\text{H}_2\text{O}]$ of an alkaloid obtained from Opium, or prepared from morphine by methylation. It should be kept in well-stoppered, amber-colored vials.

Fine, white, needle-shaped crystals, or a crystalline powder, without odor and having a bitter taste. It frequently crystallizes with one and a half molecules of water of crystallization.

Soluble in 2.25 parts of water, 261 parts of alcohol, 1340 parts of ether, and in 6620 parts of chloroform at 25° C. (77° F.); soluble in 0.46 part of water at 80° C. (176° F.) and in 97 parts of alcohol at 60° C. (140° F.).

At 100° C. (212° F.) it loses all of its water of crystallization; above 200° C. (392° F.) it becomes darker in color, and at 235° C. (455° F.) melts to a brown liquid, which rapidly volatilizes.

Its aqueous solution reddens blue litmus-paper.

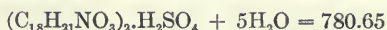
If 0.2 Gm. of Codeine Phosphate be dissolved in 5 Cc. of water and 3 Cc. of potassium hydroxide T.S., and the solution shaken out successively with three portions of chloroform (5 Cc. each), the combined chloroformic solutions, evaporated to dryness in a tared dish, should yield not less than 0.13 Gm. of codeine.

Tests for Identity.—Physical tests. See above. Tests for codeine. See Codeine. Tests for phosphate. Details in Part V.

Impurity.—Morphine. For details, see Codeine.

Assay.—Codeine, 65 per cent. Details above.

CODEINÆ SULPHAS. Codeine Sulphate.



The sulphate $[SO_2(OH)_2 \cdot (C_{17}H_{18}(CH_3)NO_3)_2 + 5H_2O]$ of an alkaloid obtained from Opium, or prepared from morphine by methylation. It should be kept in well-stoppered, amber-colored vials.

Long, glistening, white, needle-shaped crystals, rhombic prisms, or a crystalline powder efflorescing in the air, odorless and having a bitter taste.

Soluble in about 30 parts of water and 1035 parts of alcohol at 25° C. (77° F.); insoluble in chloroform and ether; soluble in 6.25 parts of water at 80° C. (176° F.) and in 340 parts of alcohol at 60° C. (140° F.).

The salt loses its water of crystallization at 100° C. (212° F.); chars, decomposes, and partially volatilizes above 200° C. (392° F.), without melting; the residue melts at about 278° C. (532.4° F.).

Its aqueous solution is neutral to litmus-paper.

Tests for Identity.—Physical tests. Given above. Tests for codeine. See Codeine. Tests for sulphates. Details in Part V.

Impurity.—Morphine. Details, see Codeine.

The uncombined alkaloid, as well as the phosphate and the sulphate, are official, and these are used as sedatives in cough-mixtures and the like, being administered in much larger doses than is morphine and its salts—one-half grain being considered as a safe dose of any of three official forms.

Dose.—Of all three, 30 milligrammes ($\frac{1}{2}$ grain).

GROUP VI.—ALKALOIDS WHOSE STRUCTURE IS LITTLE UNDERSTOOD

Physostigmine.—The structure of this alkaloid has not been sufficiently studied to make it clearly understood.

A characteristic reaction of physostigmine is the red-blue color-play produced when its ammonium hydroxide solution is evaporated on a water-bath and acidified with acetic acid; as well as the color produced with nitric acid.

The alkaloid is extracted from calabar bean by extracting same in fine powder with warm alcohol containing 0.1 per cent. tartaric acid. The alcohol is distilled from the tincture, the residue treated with water, the filtrate shaken with ether to remove color, and then made alkaline with sodium bicarbonate, and again shaken out with

ether, which this time extracts the free alkaloid, which is obtained by evaporation of the ethereal solution.

Physostigmine, or *eserine*, as it is sometimes called, is recognized by the pharmacopœia in the form of salicylate and sulphate.

PHYSOSTIGMINÆ SALICYLAS. Physostigmine Salicylate.



The salicylate $[\text{C}_6\text{H}_4(\text{OH})\text{COOH} \cdot \text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2]$ of an alkaloid obtained from Physostigma. It should be kept in small, dark amber-colored, well-stoppered vials.

Colorless or faintly yellowish, shining acicular, or short columnar crystals; odorless and having a slightly bitter taste. *It should be tasted with great caution.* It acquires a reddish tint when long exposed to light and air.

Soluble in 72.5 parts of water, 12.7 parts of alcohol, 175 parts of ether, and in 8.6 parts of chloroform at 25° C. (77° F.); soluble in 15 parts of water at 80° C. (176° F.) and in 4 parts of alcohol at 60° C. (140° F.).

When heated, Physostigmine Salicylate begins to soften and turn slightly yellow at 160° C. (320° F.) and melts at 178.9° C. (354° F.). It leaves no residue on incineration.

Its aqueous solution shows an acid reaction to litmus-paper, and upon standing twenty-four hours the solution acquires a pink color.

Ferric chloride T.S. produces, in an aqueous solution of Physostigmine Salicylate, a deep violet color; a solution of chlorinated lime added to the aqueous solution produces a red color.

On evaporating an aqueous solution of the salt to dryness with a few drops of ammonia water, a blue residue is produced, which is soluble in alcohol, and when so dissolved, yields a red fluorescent solution upon the addition of acetic acid in excess.

An aqueous solution of the salt is colored cherry-red by the addition of potassium hydroxide T.S., changing to a darker red, and finally to green.

Platinic chloride T.S. produces no precipitate in solutions of the salt (distinction from *physostigmine sulphate*).

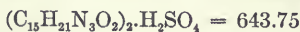
Sulphuric acid, containing in each Cc. 1 drop of solution of formaldehyde, gives with Physostigmine Salicylate a bright pink color; sulphuric acid with a few particles of cane-sugar produces a yellow color, turning to brown, then to purple, and finally to greenish-black.

If 0.005 Gm. of the salt be dissolved in nitric acid, a yellow solution results, which, on being heated, changes to an orange-color, then to blood-red, and on evaporation to dryness yields a green residue. When exposed to the fumes of nitric acid this residue becomes violet-blue, and when a drop of nitric acid is added to it, it forms a reddish-violet solution, which soon changes to blood-red, and finally, on standing, or on dilution, becomes greenish-yellow.

Tests for Identity.—Given above.

Impurity.—Physostigmine sulphate. Details above.

PHYSOSTIGMINÆ SULPHAS. Physostigmine Sulphate.



The sulphate $[\text{SO}_2(\text{OH})_2 \cdot (\text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2)_2]$ of an alkaloid obtained from Physostigma. It should be kept in well-stoppered, dark amber-colored vials.

A white or yellowish-white, micro-crystalline powder, odorless, and having a bitter taste. *It should be tasted with great caution.* It is very deliquescent and gradually turns reddish by exposure to air and light.

Very soluble in water, alcohol, and chloroform; soluble in 1200 parts of ether at 25° C. (77° F.).

When heated to 130° C. (266° F.) the salt becomes soft, and melts at about 140° C. (284° F.). Upon ignition, it is slowly consumed, leaving no residue.

Its aqueous solution shows an acid reaction to blue litmus-paper.

Barium chloride T.S. produces in an aqueous solution of the salt a white precipitate, which is insoluble in hydrochloric acid.

An aqueous solution of Physostigmine Sulphate yields, with alkalis, a white

precipitate, which quickly turns pink, and dissolves in an excess of the alkali, forming a pink red solution, which soon fades to yellowish-green.

Gold chloride T.S. gives with aqueous solutions of the salt a purple color.

Platinic chloride T.S. produces in aqueous solutions of the salt a yellowish-white precipitate (distinction from *physostigmine salicylate*).

Sulphuric acid with Physostigmine Sulphate yields only a faint yellow color.

Sulphuric acid containing a crystal of potassium iodate, on being added to a crystal of the salt, gives a light purple color, immediately changing to yellowish-red.

Tests for Identity.—Given above. Nitric acid test. See *physostigmine salicylate*.

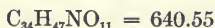
Impurity.—Physostigmine salicylate. Details above.

Remarks.—Both of these salts of physostigmine are very deliquescent, and the pharmacist is warned against buying even so small a quantity as a gramme unless the whole quantity is to be dispensed at one time; nor is it advisable to keep the physostigmine salts in solution, as the originally colorless solution turns a dark red. One of the largest manufacturers of alkaloids now dispenses physostigmine in one-grain tubes, which are sealed by a blow-pipe, thus precluding all contact with the atmosphere. Such is the only way that the pharmacist should keep eserine in stock.

Physostigmine salts are used almost entirely as miotics—that is, for producing contraction of the pupil of the eye. If given internally, they are used as tonics.

Dose.—Of both salts, 1 milligramme ($\frac{1}{84}$ grain).

ACONITINA. Aconitine.



An alkaloid obtained from Aconite. It should be kept in amber-colored, well-stoppered vials.

Colorless or white rhombic tables or prisms, possessing no odor, permanent in the air, and producing, in extremely diluted solutions, a characteristic tingling sensation when brought in contact with the mucous surfaces of the tongue or lips. *The alkaloid itself should never be tasted, and its solutions only when largely diluted, and then with the utmost caution.*

Soluble in 3200 parts of water, 22 parts of alcohol, 44 parts of ether, 5.6 parts of benzene, and 3580 parts of petroleum benzin at 25° C. (77° F.); also very soluble in chloroform.

When heated rapidly, Aconitine melts at 195° C. (383° F.); when heated slowly, decomposition takes place, and it melts at 182° C. (359.6° F.). Upon ignition, it is consumed without leaving a residue.

Aconitine solutions have an alkaline reaction upon litmus and are laevogyrate.

Aconitine, when dropped upon sulphuric or nitric acids, should produce no color, but if rubbed with sulphuric acid containing a crystal of ammonium vanadate, an orange color is produced.

Dilute solutions of Aconitine yield precipitates with mercuric potassium iodide T.S., tannic acid T.S., and gold chloride T.S., but only concentrated solutions yield precipitates with platinic chloride T.S., mercuric chloride T.S., and picric acid T.S.

On evaporating 0.01 Gm. of Aconitine with 5 drops of fuming nitric acid, the resulting yellow residue, when cooled, should not yield a violet color when treated with alcoholic potassium hydroxide T.S. (difference from *pseudaconitine* and *atropine*).

Any soluble salt of Aconitine in dilutions of 1 in 1000 produces, with a drop of potassium permanganate T.S., a blood-red precipitate of aconitine permanganate. Aconitine containing decomposition products (amorphous Aconitine) produces this precipitate only in solutions containing not less than 1 in 200. (Cocaine, hydrastine, and papaverine also yield similar precipitates, but only when in more concentrated solutions.)

Tests for Identity.—Given above.

Impurities.—Pseudoaconitine and atropine. Details above.

Extraction.—Powdered aconite is exhausted with amyl alcohol and the percolate shaken out with 1 per cent. sulphuric acid. The acid solution is freed from resin by shaking with chloroform, and is then made alkaline with sodium carbonate and shaken out with ether.

The crude alkaloid obtained by evaporation of the ethereal solution is purified by recrystallization from hydrochloric acid solution, or by making a double crystalline salt with gold chloride. In the first case the free alkaloid is obtained by treatment with alkali, while in the latter process it is freed from gold by treatment with hydrogen sulphide and then with sodium carbonate. In both cases the freed alkaloid is crystallized from ethereal solution.

Composition.—So uncertain is the composition of aconitine that the United States and British pharmacopœias assigned different empiric formulas to the alkaloid; the British claiming it has the composition $C_{33}H_{45}NO_{12}$.

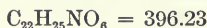
The U. S. P. formula, suggested by Freund, is now accepted; the alkaloid being, supposedly, $C_{21}H_{27}NO_3$ $\left\{ \begin{array}{l} OC_2H_5O \\ (OCH_3)_4 \\ OC_7H_5O \end{array} \right.$ that is, acetyl-benzoyl-aconine.

Remarks.—This chemical is interesting as being the most potent official substance, being administered in the smallest dose. In fact, its use internally is not advisable.

Medical Properties.—Like aconite.

Dose.—0.15 milligramme ($\frac{1}{400}$ grain).

COLCHICINA. Colchicine.



An alkaloid obtained from Colchicum. It should be kept in dark amber-colored, well-stoppered vials.

Pale yellow leaflets, or a pale yellow, amorphous powder, turning darker on exposure to light, having an odor suggesting damp hay and a very bitter taste.

Soluble in 22 parts of water, 155 parts of ether, and in 87 parts of benzene at 25° C. (77° F.); soluble in 20 parts of water at 80° C. (176° F.); very soluble in alcohol and chloroform; insoluble in petroleum benzin.

After drying over sulphuric acid, it melts if heated to 142.5° C. (288.5° F.). It leaves no residue upon incineration.

Its aqueous solution is neutral to litmus-paper, lævogyrate, and of a yellow color, which is intensified by mineral acids.

With Colchicine, sulphuric acid produces a citron-yellow color, which, upon adding a drop of nitric acid, changes to greenish-blue, then to red, and finally to yellow. On adding an excess of potassium hydroxide, the color is changed to red.

Ferric chloride T.S., on being added to an aqueous solution of Colchicine, gives no color, but on heating a brownish-red color is developed, which changes to brownish-black; if ferric chloride T.S. be added to an alcoholic solution of Colchicine, a garnet-red color is at once produced.

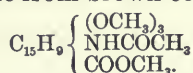
To 5 drops of an aqueous Colchicine solution add 5 drops of fuming hydrochloric acid, 5 drops of ferric chloride T.S., and then heat to boiling, when a yellow solution will be formed, changing to olive-green. When cool, shake the liquid with a little chloroform, when the latter will turn ruby-red, and the aqueous solution will remain green.

Sulphuric acid containing a fragment of potassium dichromate gives a greenish-blue color, changing to orange.

Tests for Identity.—Given above.

Extraction.—Powdered colchicum seed are extracted with 90 per cent. hot alcohol, the alcohol recovered by distillation, and the residue treated with water. The filtrate is shaken out with chloroform, the chloroformic solution of the alkaloid evaporated, the residue dissolved in water, and again shaken out with chloroform. On evaporation of the solvent a crystalline chloroformic compound of colchicine is left, and this is dissolved in alcohol, concentrated by distillation, again shaken out with chloroform, and the chloroform distilled off. The residue is dissolved in warm ether, and the colchicine-chloroform crystals mixed with water and warmed until the chloroform evaporates. The aqueous solution of purified colchicine is then evaporated to dryness. The shaking out with several portions of chloroform is necessary to free the colchicine from brown coloring-matter.

Composition.—



Medical Properties.—Like colchicum (p. 919).

Dose.— $\frac{1}{2}$ milligramme ($\frac{1}{128}$ grain).

PELLETIERINÆ TANNAS. Pelletierine Tannate.

A mixture in varying proportions of the tannates of four alkaloids (punicine, iso-punicine, methyl-punicine, and pseudo-punicine) obtained from *Punica Granatum* Linné (Fam. *Punicaceæ*). It should be kept in small, well-stoppered, dark amber-colored vials.

A light yellow, odorless, amorphous powder, having an astringent taste and a weak acid reaction.

Soluble in 235 parts of water, 12.6 parts of alcohol, and in 300 parts of ether at 25° C. (77° F.); insoluble in chloroform; soluble in warm diluted acids.

Pelletierine Tannate, when dried over sulphuric acid and heated, turns brown at 150° C. (302° F.), softens at about 165° C. (329° F.), and when heated to a higher temperature, decomposes and chars without melting. It leaves no residue on ignition.

Ferric chloride T.S. colors aqueous solutions of the salt blue-black.

In aqueous solutions of Pelletierine Tannate, soluble lead, mercury, and zinc salts produce white precipitates.

Platinic chloride T.S. produces no precipitate.

Ammonia water produces a white precipitate, soluble in excess of the precipitant, forming a yellowish-red solution.

Aqueous solutions of Pelletierine Tannate immediately reduce silver nitrate T.S., or gold chloride T.S., to metallic silver or gold respectively, the former as a black precipitate, and the latter as a thin, purplish mirror on the test-tube.

Sulphuric acid gives a yellow color; the liquid, on being heated, turns slowly to green, and finally to purple. Nitric acid produces no color.

Sulphuric acid containing a trace of selenous acid produces a light bluish-green color, gradually becoming dark green, and developing a pink border.

Tests for Identity.—Given above.

Manufacture.—Ground pomegranate bark is mixed with milk of lime and extracted with water. The percolate is shaken with chloroform and the separated chloroformic layer is shaken with diluted sulphuric acid. The acid solution of the sulphates of the alkaloids is treated with tannic acid and the precipitated tannates collected on a filter and dried.

Composition.—As mentioned in the official definition given above, this body is a mixture of four alkaloids. The free alkaloidal mixture

is a volatile liquid, but the official tannate, like all salts of liquid alkaloids, is a solid.

Medical Properties.—Tenifuge.

Dose.—250 milligrammes (4 grains).

GROUP VII.—ALKALOIDAL DRUGS

IPECACUANHA. *Ipecac.*

The dried root, to which may be attached a portion of the stem not exceeding 7 Cm. in length, of *Cephaelis Ipecacuanha* (Brotero) A. Richard (Fam. *Rubiaceæ*), known commercially as *Rio*, *Brazilian*, or *Para ipecac*, or the corresponding portion of *C. acuminata* Karsten, known commercially as *Carthagera ipecac*, yielding, when assayed by the process given on p. 1033, not less than 1.75 per cent. of ipecac alkaloids.

Assay.—Part V. (p. 1033).

Constituents.—*Emetine*, *cephaeline*, *psychotrine*, *ipecacuanhic acid* (a form of tannin), resin, starch, etc.

Medical Properties.—Expectorant or emetic, according to dose. Official preparations: *Fluidextract* (p. 246) and *Dover's powder* (p. 304). The fluidextract is a constituent of the *syrup* (p. 196), the *wine* (p. 259), and *tincture of ipecac and opium* (p. 228).

Dose.—Of drug: Expectorant, 65 milligrammes (1 grain); emetic, 1 Gm. (15 grains).

GELSEMIUM. *Gelsemium*. Yellow Jasmin.

The dried rhizome and roots of *Gelsemium sempervirens* (Linné) Aiton filius (Fam. *Loganiaceæ*).

Constituents.—*Gelsemine*, *gelseminine*, *æsculin* (called originally *gelseminic acid*), volatile oil, resin, etc.

Medical Properties.—Antipyretic, etc. The *tincture* (p. 227) and *fluidextract* (p. 246) are official.

Dose.—65 milligrammes (1 grain).

PAREIRA. *Pareira*.

The dried root of *Chondrodendron tomentosum* Ruiz et Pavon (Fam. *Menispermaceæ*).

Constituents.—*Cissampeline*, fat, starch, and tannin.

Medical Properties.—Simple bitter, diuretic. The *fluidextract* (p. 248) is official.

Dose.—2 Gm. (30 grains).

ACONITUM. *Aconite*.

The dried tuberous root of *Aconitum Napellus* Linné (Fam. *Ranunculaceæ*), collected in autumn; yielding, when assayed by the process given on p. 1027, not less than 0.5 per cent. of aconitine.

Assay.—Part V. (p. 1027).

Constituents.—*Aconitine*, *napelline*, *aconitic acid*, etc. *Aconitine* consists of true aconitine, pseudaconitine, aconine, pseudaconine, piraconitine, etc.

Remarks.—*Aconite* is a tuberous root from the *Ranunculaceæ*, or crowfoot family. Its well-known poisonous property is due to its

alkaloid, aconitine, which is supposed to exist in the root as the salt of aconitic acid. This acid is closely allied to citric acid, as a side-by-side comparison formula will show:



Like citric acid, aconitic acid is not poisonous. It is found in several plants other than aconite; for instance, sorghum and yarrow.

Medical Properties.—Of value in fevers produced from colds, neuralgia, etc. Locally as a local irritant and anesthetic. The tincture (p. 221) and *fluidextract* (p. 240) are official.

Dose.—65 milligrammes (1 grain).

BERBERIS. Berberis. Oregon Grape Fruit.

The rhizome and roots of *Berberis Aquifolium* Pursh, and other species of *Berberis* (Fam. *Berberidaceæ*).

Constituents.—Berberine, $\text{C}_{17}\text{H}_{20}\text{NO}_4$ (see p. 905); oxyacanthine, $\text{C}_{19}\text{H}_{21}\text{NO}_3$; berbamine, $\text{C}_{18}\text{H}_{19}\text{NO}_3$.

Remarks.—This new official is not obtained from the ordinary barberry bush (*Berberis vulgaris*), but is derived from that species growing in the Western States. The *fluidextract* is recognized by the present pharmacopœia.

Medical Properties.—Tonic, stimulant, antiperiodic, and hepatic stimulant.

Dose.—2 Gm. (30 grains).

COLCHICI CORMUS. Colchicum Corm.

[COLCHICI RADIX, PHARM. 1890]

The dried corm of *Colchicum autumnale* Linné (Fam. *Liliaceæ*), yielding, when assayed by the process given on p. 1019, not less than 0.35 per cent. of colchicine.

Constituents.—Colchicine, colchiceine, and two resins.

Remarks.—*Colchicum* is official in two forms: the "corm" and the seed of *Colchicum autumnale*, a pretty little liliaceous plant called "meadow-saffron," that decks the pastures of South Germany with beautiful pink flowers during the month of September. A "corm" is a variety of tuber, the most familiar example of a tuber being the Irish potato. The potato grows underground, but it is not a root, for, bear in mind, that is an erroneous impression that an underground plant part has to be a root. The difference between a root and a stem is that while the latter *always* bears leaves, the former *never* does. The potato must, therefore, be a stem part, for it bears leaves, the "eyes" of the potato being minute leaf groups, which sprout under favorable circumstances. Such a thick, fleshy stem part, growing underground and designed as the storehouse for food for the next year's growth, is called a tuber, and colchicum root is a modification

of a tuber called a corm. A corm differs from a tuber by growing at the immediate base of the stem instead of at the end of a subterranean shoot, as the regular tuber does.

Medical Properties.—Of great value in gout. Its use, however, must not be long continued, as it soon irritates the stomach and intestines.

The *extract* (p. 281) is official.

Dose.—Of drug, 250 milligrammes (4 grains).

COLCHICI SEMEN. Colchicum Seed.

The seed of *Colchicum autumnale* Linné (Fam. *Liliaceæ*), yielding, when assayed by the process given on p. 1020, not less than 0.55 per cent. of colchicine.

Assay.—See p. 1020.

Constituents.—Like the corm, only less colchicine and 6 per cent. fixed oil.

Medical Properties.—Like the corm. The *Wine* (p. 257), *tincture* (p. 226), and *fluidextract* (p. 247) are official.

Dose.—200 milligrammes (3 grains).

SANGUINARIA. Sanguinaria. Blood Root.

The dried rhizome of *Sanguinaria canadensis* Linné (Fam. *Papaveraceæ*), collected after the death of the foliage.

Constituents.—*Sanguinarine*, *chelerythrine*, *protopine*, *berberine*, resins, etc.

Remarks.—The drug is called blood root because, in the spring, the fresh rhizome, when broken, exudes a red juice. Likewise the dried drug is deep red in the interior.

Medical Properties.—Nauseant expectorant. The *tincture* (p. 235) and the *fluidextract* (p. 251) are official.

Dose.—125 milligrammes (2 grains).

SPIGELIA. Spigelia. Pink Root.

The dried rhizome and roots of *Spigelia marilandica* Linné (Fam. *Loganiaceæ*).

Constituents.—*Spigeline* (volatile alkaloid), volatile oil, resins, etc.

Medical Properties.—Vermifuge. Administered as *fluidextract*, U. S. P. (p. 247).

Dose.—4 Gm. (60 grains).

HYDRASTIS. Hydrastis. Golden Seal.

The dried rhizome and roots of *Hydrastis canadensis* Linné (Fam. *Ranunculaceæ*), yielding, when assayed by the process given on p. 1024, not less than 2.5 per cent. of hydrastine.

Constituents.—*Hydrastine*, *berberine*, *canadine*, resin, starch, etc.

Remarks.—Hydrastis, the rhizome and rootlets of *Hydrastis canadensis*, is an American drug, which has obtained world-wide reputation. Note its two leading constituents, of which the hydrastine should be white. Hydrastine is very expensive, hence the cheap yellow berberine is often substituted for it. Note that it is berberine

which gives the drug the characteristic yellow color, which suggested the synonym, golden seal.

Medical Properties.—Bitter stomachic, useful in catarrhal trouble, hence of value as injection in gonorrhea. For this purpose the official *glycerite* (p. 207) is largely used. The *tincture* (p. 228) and the *fluidextract* (p. 248) are also official.

Dose.—2 Gm. (30 grains).

VERATRUM. Veratrum. Hellebore.

[VERATRUM VIRIDE, PHARM. 1890]

The dried rhizome and roots of *Veratrum viride* Aiton (American Hellebore) or *Veratrum album* Linné (White Hellebore) (Fam. *Liliaceæ*).

Constituents.—*Jervine*, *pseudojervine*, *rubijervine*, *veratroidine*, *cevadine*. White veratrum contains veratrine and also the alkaloids above mentioned. The official veratrine comes from cevadilla seed (p. 903).

Remarks.—In the pharmacopœia of 1890 only the American hellebore was official, but in the last revision the white hellebore of Europe was admitted, since much of it finds its way into the American market, and since the two are therapeutically identical.

CINCHONA. Cinchona. Peruvian Bark. Calisaya Bark.

The dried bark of *Cinchona Ledgeriana* Moens, *Cinchona Calisaya* Weddell, *Cinchona officinalis* Linné, and of hybrids of these with other species of *Cinchona* (Fam. *Rubiaceæ*). It should yield not less than 5 per cent. of total anhydrous cinchona alkaloids and at least 4 per cent. of anhydrous ether-soluble alkaloids when assayed by the process given on p. 1018.

CINCHONA RUBRA. Red Cinchona. Red Bark.

The dried bark of *Cinchona succirubra* Pavon (Fam. *Rubiaceæ*), or of its hybrids, yielding not less than 5 per cent. of anhydrous cinchona alkaloids when assayed by the process given for these alkaloids on p. 1018.

Constituents.—Quinine, quinidine, cinchonine, cinchonidine, and many other alkaloids, kinic acid, kinovic acid, and cinchotannic acid.

Cinchona barks are alkaloidal drugs, whose value is scarcely second to opium. In these barks the pharmacopœia gives a wide range of selection, for, while dividing them into two groups,—*cinchona* and *cinchona rubra*,—that authority allows the use of any cinchona bark containing 5 per cent. of the alkaloids peculiar to the species. Under the heading "*cinchona rubra*" we find but one species allowed—*Cinchona succirubra*, the last word meaning "red juice." but under the head "*cinchona*" permission is given to use any cinchona yielding 5 per cent. of total alkaloids and 4 per cent. of anhydrous ether-soluble alkaloids.

Time forbids an extended sketch of the origin and history of the cinchona barks, but the student is advised to read the subject in the dispensaries. Here need only be said that the general introduction of the bark was due to the Countess of Chinchon, wife of a viceroy of Peru, who was cured of malaria by the use of the drug in the sixteenth century, since when it has gained the esteem of the entire world.

The reckless collection of the drug by chopping down wild trees, with no attempt to plant new ones in their stead, threatened the extermination of this valuable drug, but, fortunately, this danger was averted by the commencement of the systematic cultivation of the drug by the Dutch in Java and by the English in India, who have taken most of the business from its first home—Chile and Peru.

Note that of the large number of alkaloids separated from cinchona, but four—quinine, quinidine, cinchonine, cinchonidine—are worthy of careful study.

Note also that cinchona contains a large amount of tannin, hence glycerin is added to the menstruum in preparing its official preparations. This also explains the well-known prescription incompatibility, the inky liquid obtained by mixing preparations of cinchona with iron salts (p. 1078); hence in preparing such pharmaceuticals as elixir of cinchona and iron the cinchona percolate must be “detannated” before adding the iron salt.

Mention must be made of a former official product of cinchona, which was dropped in the revision of 1890; a substance called *chinoidine*. In the days when quinine was worth \$4 to \$5 an ounce, a cheaper cinchona product was demanded. This was found in chinoidine, whose efficiency was due to the imperfect methods then employed in making quinine. Quinine is separated from the other cinchona alkaloids by crystallization, and in the old process, after the mother liquor had been concentrated several times, thus yielding most of the four official alkaloids, it was evaporated to a brown mass, still containing some of the four alkaloids, as well as all the minor bodies found in the bark. But the old-time primitive methods of extraction have given place to modern processes, by which almost all of the important alkaloids are won from the cinchona, leaving the mother liquor practically inert. Hence chinoidine of to-day is almost valueless and in the pharmacopœial revision of 1890 it was omitted.

Medical Properties.—Febrifuge (quinine is a specific in malarial fever), tonic astringent. Official preparations: Of red bark, *compound tincture* (p. 226), of yellow bark, *tincture* (p. 225) and *fluid-extract* (p. 248).

Dose.—Of both barks, 1 Gm. (15 grains).

GRANATUM. Pomegranate.

The bark of the stem and root of *Punica Granatum* Linné (Fam. *Punicacæ*).

Constituents.—*Pelletierine*, U. S. P., punicotannic acid.

Remarks.—Note that the official drug is the bark of the stem and root, not the rind of the fruit, which is so popular in the southern States as a dysentery remedy.

Medical Property.—Tenifuge. Administered preferably as the alkaloid pelletierine, or as *fluidextract*, U. S. P. (p. 248).

Dose.—Of drug, 2 Gm. (30 grains).

Quebracho, the bark of *Aspidosperma Quebracho-blanco*, was recognized by the pharmacopœia of 1890, but was dropped in the last revision. It contains some alkaloids and much tannin, and was supposed to be a bitter tonic.

Dose.—4 Gm. (60 grains).

PILOCARPUS. Pilocarpus. Jaborandi.

The leaflets of *Pilocarpus Jaborandi* Holmes or of *Pilocarpus microphyllus* Stapf (Fam. *Rutacæ*), yielding, when assayed by the process given on p. 1038, not less than 0.5 per cent. of alkaloids.

Assay.—See Part V. (p. 1038).

Constituents.—*Pilocarpine*, *jaborine*, *pilocarpidine* (an oxidation product of pilocarpine), *jaboridine* (an oxidation product of jaborine).

Medical Properties.—Valuable diaphoretic and sialagogue. Administered in the form of the official *fluidextract* (p. 247).

Dose.—2 Gm. (30 grains).

COCA. Coca.

The dried leaflets of *Erythroxylon Coca* Lamarek (Fam. *Erythroxylaceæ*), known commercially as Huanuco Coca, or of *E. Truxillense* Rusby, known commercially as Truxillo Coca, yielding, when assayed by the process given on p. 1031, not less than 0.5 per cent. of the ether-soluble alkaloids of Coca.

Constituents.—Cocaine, hygrine, cocatannic acid.

Remarks.—This drug has been used by the natives of Peru since the earliest times as a stimulant. The Indians chew the leaves mixed with ashes, and are then capable of performing such hard tasks as climbing mountains with loaded backs, practically abstaining from food during the trip. Its use as a tonic in Europe and North America covers a period of many years, but its great consumption began in the early eighties, with the discovery of the remarkable local anesthetic action of its alkaloid, cocaine, made official in 1890 in the form of *cocaine hydrochlorate*. The value of cocaine in surgery and the frightful effects of the abuse of the chemical are too well known to require extended comment here.

Medical Properties.—Stimulant. The *wine* (p. 257) and *fluid-extract* (p. 247) are official.

Dose.—2 Gm. (30 grains).

BELLADONNÆ FOLIA. Belladonna Leaves. Deadly Nightshade.

The dried leaves of *Atropa Belladonna* Linné (Fam. *Solanaceæ*), yielding, when assayed by the process given on p. 1030, not less than 0.30 per cent. of mydriatic alkaloids.

Constituents.—*Atropine*, *hyoscyamine*, *belladonnine*, *hyoscine*, and other alkaloids, chlorophyll, etc.

Remarks.—Belladonna is derived from the "deadly nightshade," *Atropa Belladonna*, the leaves and root of which are official. Both contain atropine, which dilates the pupil of the eye, making it large and lustrous. From this property the drug gets its name, which is Italian for "pretty lady." Besides atropine, the drug contains two other official alkaloids, hyoscyamine and hyoscine, these two being, of course, named after hyoscyamus, in which they are first found. All three of these alkaloids are found in various proportions in many of the plants of the *Solanaceæ*—the nightshade family—to which both belladonna and hyoscyamus belong, and all three, by reason of the property of dilating the pupil, are called the *mydriatic alkaloids*. Hyoscyamine and atropine are isomeres—both have the same chemical formula. In fact, so similar are they that they can be distinguished only by the differing behavior of their gold salts.

Medical Properties.—Stimulates the higher centers, hence used in asthma and to quicken the pulse; used to modify the action of vege-

table purgatives, as is shown in the official *compound laxative pills*, in which extract of belladonna leaves is employed. Used externally in neuralgia. Official preparations: the *extract* (p. 279) and the *tincture* (p. 223). From the extract the *ointment* (p. 345) and the *plaster* (p. 355) are made.

Dose.—65 milligrammes (1 grain).

BELLADONNÆ RADIX. Belladonna Root.

The dried root of *Atropa Belladonna* Linné (Fam. *Solanaceæ*), yielding, when assayed as directed on p. 1031, not less than 0.45 per cent. of mydriatic alkaloids.

Constituents.—Like the leaves, except for absence of chlorophyll.

Remarks.—Note that the root contains more mydriatic alkaloids than do the leaves (root, $\frac{4.5}{100}$ of 1 per cent.; leaves, $\frac{3}{10}$ of 1 per cent.), hence it is administered in smaller dose.

Medical Properties.—Like the leaves. The *fluidextract* (p. 246) is official, and from it is made the official *liniment* (p. 272).

Dose.—Of drug, 45 milligrammes ($\frac{3}{4}$ grain).

STRAMONIUM. Stramonium. Jimson Weed. Jamestown Weed. Thornapple.

[STRAMONIUM FOLIA, PHARM. 1890]

The dried leaves of *Datura Stramonium* Linné (Fam. *Solanaceæ*), yielding, when assayed as directed on p. 1039, not less than 0.25 per cent. of mydriatic alkaloids.

Constituents.—Atropine, hyoscyne, hyoscyamine, chlorophyll. The constituent *daturine* has been proved a mixture of atropine and hyoscyamine.

Remarks.—Stramonium is the leaves of *Datura Stramonium*, which is the well-known “jimson weed,” a plant growing all over the United States, Europe, and Asia. It is said that the plant has become so widely distributed through those curious people, the gypsies, who travel all over the world, carrying with them stramonium seed—for criminal uses it is said. Note that stramonium belongs to that plant family called the *Solanaceæ*, or nightshade family, which furnishes such valuable drugs as belladonna and hyoscyamus, and also includes several of our best-known vegetables, like Irish potato, tomato, and egg-plant.

Belladonna, hyoscyamus, and stramonium contain the three mydriatic alkaloids, atropine, hyoscyne, and hyoscyamine. Quite often we find such chemical similarities existing between members of the same plant family—a very striking fact when we consider that the method by which we classify plants has nothing to do with chemistry.

Medical Properties.—Like belladonna. The *extract* (p. 289), the *tincture* (p. 236), and the *fluidextract* (p. 247) are official. From the extract the official *ointment* (p. 349) is prepared.

Dose.—65 milligrammes (1 grain).

Stramonium seeds, which were recognized by the pharmacopœia of 1890, never enjoyed the same popularity as the leaves, hence were dropped in the last revision. They contained the same alkaloids and were used similarly to the leaves and given in the same dose.

HYOSCYAMUS. Hyoscyamus. Henbane.

The dried leaves and flowering tops of *Hyoscyamus niger* Linné (Fam. *Solanaceæ*), collected from plants of the second year's growth, and yielding, when assayed as directed on p. 1032, not less than 0.08 per cent. of mydriatic alkaloids.

Constituents.—Hyoscyamine and hyoscyine.

Remarks.—Hyoscyamus is commonly called henbane, the official drug being leaves plucked from the second-year stem, those of the first year being small and almost inert. Note that the drug is much weaker in mydriatic alkaloids than are belladonna and stramonium leaves, the last two being required to contain $\frac{3.5}{100}$ of 1 per cent. alkaloids, while the requirement for hyoscyamus is but $\frac{8}{100}$ of 1 per cent.

Medical Properties.—Similar to belladonna, but preferable to the latter drug in insomnia and for relieving the excitement of insanity. Official preparations: *Extract* (p. 284), *fluidextract* (p. 247), and *tincture* (p. 228). The fresh leaves steeped in a fixed oil along with other drugs and aromatics is highly popular in Europe under the name *Baume Tranquille*, as a remedy for earache and for general use as a liniment. A similar preparation is the *compound oil of hyoscyamus*, N. F.

Dose.—Of drug, 250 milligrammes (4 grains).

SCOPOLA. Scopola.

The dried rhizome of *Scopola Carniolica* Jacquin (Fam. *Solanaceæ*), yielding, when assayed as directed on p. 1039, not less than 0.5 per cent. of mydriatic alkaloids.

Remarks.—This new official contains *scopolamine*, an alkaloid identical with hyoscyine. The drug itself is rarely used, and has been recognized chiefly because used for making scopolamine, and because its extract is used by some plaster-makers for their so-called belladonna plasters. If used, it is, like belladonna and hyoscyamus, a sedative and narcotic.

Dose.—45 milligrammes ($\frac{3}{4}$ grain).

Tobacco, the leaves of *Nicotiana tabacum*, was official in former pharmacopœias, but was dropped in the last revision. It was formerly highly esteemed as a vulnerary, but is little used as a drug by intelligent physicians. A decoction of tobacco in which corrosive sublimate has been dissolved makes a satisfactory *bedbug poison*.

LOBELIA. Lobelia.

The dried leaves and tops of *Lobelia inflata* Linné (Fam. *Campanulaceæ*), collected after a portion of the capsules have become inflated.

Constituents.—*Lobeline* (a liquid alkaloid), *lobelic acid*, resin, etc.

Remarks.—The pharmacopœial requirement, given above, as to “inflated capsules,” means that the drug must be collected only when completely ripe. The fruit of the lobelia is a round pod, consisting of minute brown seeds inclosed in a thin, papery “husk,” which swells considerably when seeds are ripe. This enlarged pod is what the definition means by “inflated capsule.”

Medical Properties.—Expectorant and emetic. Poisonous in large doses. The *tincture* (p. 231) and the *fluidextract* (p. 250) are official.

Dose.—0.5 Gm. ($7\frac{1}{2}$ grains).

SCOPARIUS. Scoparius. Broomtop.

The dried tops of *Cytisus Scoparius* (Linné) Link (Fam. *Leguminosæ*).

Constituents.—*Sparteine* (liquid alkaloid), *scoparin* (neutral principle), volatile oil, tannin, etc.

Remarks.—Scoparius, or broom, is the “*planta genista*,” from which the famous Plantagenet family took its name. It is not the broom-straw from which the modern American brooms used in sweeping are made, albeit a certain druggist was once known to fill a prescription for “decoction of broom” from the straw of that homely domestic article.

Medical Properties.—Diuretic (due to scoparin, rather than to sparteine). Slows the heart, but also weakens it.

Dose.—1 Gm. (15 grains).

Chelidonium.—The entire plant, *Chelidonium majus*, was official in the pharmacopœia of 1890. It, like the plant yielding opium, belongs to the natural order Papaveraceæ, the poppy family, and its juice also is somewhat narcotic. The drug is not used very largely. To the poppy family belongs also sanguinaria.

CONIUM. Conium. Hemlock.

The full-grown, but unripe fruit of *Conium maculatum* Linné (Fam. *Umbelliferae*), carefully dried and preserved, and yielding, when assayed by the process given on p. 1022, not less than 0.5 per cent. of coniine. After being kept for more than two years Conium is unfit for use.

Constituents.—Coniine, trace of volatile oil, fixed oil, etc.

Remarks.—Conium, like anise, coriander, fennel, and caraway, is a fruit of the natural order Umbellifera, and closely resembles anise in appearance—so much so that it has been confused with that drug, and with serious results. Unlike the four fruits just mentioned, it contains little or no aromatic oil, and does contain the poisonous liquid alkaloid coniine. This alkaloid has the peculiar odor of mice’s urine, thus affording a good method of distinguishing conium from anise. Conium is that “seed,” or, more correctly, that fruit, which, when triturated with solution of soda, gives off the odor of mice’s urine.

Medical Properties.—Narcotic, sedative, and antiseptic. The *fluidextract* (p. 248) is official.

Dose.—200 milligrammes (3 grains).

NUX VOMICA. Nux Vomica. Dog Button.

The dried ripe seed of *Strychnos Nux-vomica* Linné (Fam. *Loganiaceæ*), yielding, when assayed by the process given on p. 1035, not less than 1.25 per cent. of strychnine.

Constituents.—*Strychnine*, *brucine*, *igasuric acid*, *loganine*, fat, and albumin.

Remarks.—The “buttons” of *nux vomica* are very characteristic, and their silky appearance is due to the fine hairs with which they are covered. The fixed oil sometimes proves troublesome pharmaceutically, the alcoholic preparations frequently turning milky on addition of water.

Medical Properties.—Nervous stimulant and bitter tonic. Very poisonous. Antidote is *not* chloral hydrate, which merely lessens the force of the tetanic convulsions, and should be used only under direct supervision of the physician. The correct antidote is an alkaloidal precipitant, such as tannin, compound solution of iodine, or potassium permanganate. The *extract* (p. 286), *tincture* (p. 231), and *fluid-extract* (p. 249) of *nux vomica* are official.

Dose.—65 milligrammes (1 grain).

PHYSOSTIGMA. Physostigma. Calabar Bean.

The ripe seed of *Physostigma venenosum* Balfour (Fam. *Leguminosæ*), yielding, when assayed by the process given on p. 1037, not less than 0.15 per cent. of alkaloids soluble in ether.

Constituents.—*Physostigmine* (or *eserine*), *calabarine*, *eseridine*, *physosterin* (a form of proteid).

Remarks.—Physostigmine is called “Calabar bean,” from that section of Africa where it grows. Its alkaloid, physostigmine, is official as the sulphate and as the salicylate, which is less deliquescent than the other salts (p. 915).

Medical Properties.—Similarly to strychnine, physostigmine (p. 915) is used for contracting the pupil of the eye—is a miotic, just the reverse of a mydriatic (p. 923). For this reason miotic drugs (like strychnine and physostigmine) have been suggested as antidotes to mydriatics (such as belladonna and hyoscyamus), and similarly mydriatics have been used as antidotes to miotics. Such reasoning is not, however, necessarily logical. The *extract* (p. 287) and *tincture* (p. 234) of physostigma are official.

Dose.—100 milligrammes (1½ grains).

STAPHISAGRIA. Staphisagria. Stavesacre.

The ripe seed of *Delphinium Staphisagria* Linné (Fam. *Ranunculaceæ*).

Constituents.—*Delphinine*, *delphinoidine*, *staphisagrin*, the latter a mixture.

Remarks.—Staphisagria is the pharmacopœial name for the seeds of *Delphinium Staphisagria*, a plant closely related to the garden larkspur, *Delphinium consolida*. The former plant has odd-shaped flowers resembling the larkspur. Stavesacre, as the official drug is called, is a very potent remedy, and is used chiefly as a wash for body-lice.

Medical Properties.—When used internally it acts something like aconite. Rarely employed, however, except externally (see above). The *fluidextract* (p. 246) is official.

Dose.—65 milligrammes (1 grain).

ERGOTA. Ergot.

The sclerotium of *Claviceps purpurea* (Fries) Tulasne (Fam. *Hypocreaceæ*), replacing the grain of rye, *Secale cereale* Linné (Fam. *Gramineæ*). Ergot should be moderately dried and not exposed to a damp atmosphere. After being kept more than one year it is unfit for use.

Constituents.—*Cornutine* (or *ergotinine*), $C_{35}H_{39}O_5N_5$; *ergotoxine*, $C_{35}H_{41}O_6N_5$, the hydroxide of ergotinine, claimed by Barker and Dale to be the most active principle of ergot; *ergotinic acid*. *Cornutine* is supposed to be the *ecboline* of Wenzell, ergotoxine is apparently the *sphacelinic acid* of Kobert; while ergotinic acid is now considered the *sclerotic acid* of Dragendorff.

Remarks.—The constituents of fungi are classed among the most difficult problems of organic chemistry, hence it is not strange that there has been such diversity in the reports of investigations of ergot.

Ergot is a very important drug, whose official definition requires explanation, and this means a discussion of the life-history of an interesting representative of the non-flowering plants or cryptogams. Referring to the official definition, we find that ergot is "the sclerotium of *Claviceps purpurea*, . . . replacing the grain of rye, *Secale cereale*." As to "the grain of rye," that plant is a cultivated grass, used for food, similar to wheat or rice, and its fruit, a long cluster of oval grains, each protected by a chaffy cover, is familiar to most of us. Each grain was, in its early stage, a tender ovary, filled with soft and nutritious tissue. At this stage of growth it is apt to be attacked by the spores of the fungoid plant, *Claviceps purpurea*; the spores of non-flowering plants answering to the seed of flowering plants, an official example of which is lycopodium, noted on (p. 736). These spores of *Claviceps purpurea* float through the air, seeking a location where they can obtain nourishment with as little work as possible; for the plant, *Claviceps purpurea*, is one of the "tramps" of botany—a parasite which does not work into food the materials obtained from the earth and air (as do all "good" plants), but which lazily fastens its roots into soft tissue of some living and thrifty plant and sucks from the latter, nutriment stored up for personal use.

In this manner the spores of *Claviceps purpurea* light upon the tender ovaries of the young rye, *Secale cereale*. As seed planted in goodly ground begins to sprout, so do the spores of ergot sprout when they drop upon the rye. The rootlets of each spore penetrate into an ovary of the rye from which they suck nutriment and increase in size, at the expense of their host, until eventually, from what was originally a minute fungus growth (called the mycelium), the ergot, literally eating the ovary of the rye "out of house and home," develops into a brown purple fusiform growth larger than the remaining rye grains, called, by reason of its hardness, the sclerotium. This sclerotium constitutes the official drug—ergot. A word as to the future growth of the sclerotium, which is a provision of nature for the preservation of the fungus over winter. It reaches full growth in early fall and drops to the earth, where, owing to its color, it is apt to lie unnoticed over winter—apparently a dead thing. But the warmth and sunshine of spring awakens the spark of life and it then puts forth the fruit head in which are formed the spores, which are again

wafted into the air to begin once more the history of another year's havoc of the rye.

Ergot is a potent remedy of inestimable value in checking hemorrhage and in childbirth. The rye mixed with ergot is of course dangerous, and on several occasions entire communities have been poisoned by eating bread made from rye flour containing ergot.

Medical Properties.—Used as an ecbotic, producing contraction of the uterus, and for hemorrhages. Official preparation, the *fluid-extract* (p. 249), and from this the official *extract* (p. 282) and *wine* (p. 258) are made.

Dose.—2 Gm. (30 grains).

OPIUM. Opium.

The concrete, milky exudation obtained by incising the unripe capsules of *Papaver somniferum* Linné (Fam. *Papaveraceæ*), and yielding, in its normal, moist condition, not less than 9 per cent. of crystallized morphine when assayed by the process given on p. 1025.

OPII PULVIS. Powdered Opium.

Opium dried at a temperature not exceeding 85° C. (185° F.) and reduced to a very fine powder.

Powdered Opium, for pharmaceutic and medicinal purposes, when assayed by the process given on p. 1025, should yield not less than 12 per cent. nor more than 12.5 per cent. of crystallized morphine.

Powdered Opium of a higher percentage may be brought within these limits by admixture with Powdered Opium of a lower percentage or powdered sugar of milk in proper proportions.

OPII GRANULATUM. Granulated Opium.

Opium dried at a temperature not exceeding 85° C. (185° F.) and reduced to a coarse (No. 20) powder.

Granulated Opium, when assayed by the process given on p. 1025, should yield not less than 12 per cent. nor more than 12.5 per cent. of crystallized morphine. Granulated Opium of a higher percentage may be brought within these limits by admixture with Granulated Opium of a lower percentage in proper proportions.

Constituents.—*Morphine*, *codeine*, *narceine* *narcotine*, and many other alkaloids, meconic acid and lactic acid gum, etc. No starch.

Remarks.—Read carefully the pharmacopœial definition given above, and note that opium is “the concrete milky exudation” obtained by cutting—“incising”—the unripe capsules of *Papaver somniferum*. The poppy capsules (or heads) found in drug stores are from the same plant, but are collected on ripening, when they do not contain so much milk juice.

An analogous case is found in the fig, which abounds in milk juice when green and has scarcely any when ripe.

Papaver somniferum can grow as well in this country as it can in Asia Minor, and the only reason why opium is not produced here is because the incising of the capsule (slitting with a sharp knife without penetrating the capsule) is a laborious task performed by hand, and in such work America cannot compete with the cheap labor of the East. Note that the three official forms of opium given above, the

moist gum, containing not less than 9 per cent. of morphine, the *powdered*, and the *granulated*, prepared by driving off the moisture from the gum and converting the dried mass either to powder or to granules, the latter made by moistening with alcohol and rubbing through a coarse sieve.

Both the powdered and the granulated must contain not less than 12 per cent. nor more than $12\frac{1}{2}$ per cent. of crystallized morphine.

Medical Properties.—Sedative and narcotic. Poisonous. *Antidote* stimulants (caffeine or coffee), electric shocks, flagellation. Above all, keep patient in motion. Official preparations: From the *moist gum* we make the *powdered* and the *granulated*; from the *powdered* we make *deodorized opium* (see below), the *camphorated tincture* (p. 233), the *extract* (p. 286), the *pills* (p. 324), the *vinegar* (p. 263), and *powder of ipecac and opium* (p. 304), *lozenges of glycyrrhiza and opium* (p. 332); from the *granulated* we make the *tincture* (p. 232), the *deodorized tincture* (p. 233), and the *wine* (p. 259)—in short, the three preparations of opium made by percolation; lastly, the extract is an ingredient of *opium plaster* (p. 356).

Dose.—Of the *moist gum*, 100 milligrammes ($1\frac{1}{2}$ grains); of the *powdered* or *granulated*, 65 milligrammes (1 grain).

OPIUM DEODORATUM. Deodorized Opium.

Recipe.—Powdered Opium, five hundred grammes..... 500 Gm.
Purified Petroleum Benzin, a sufficient quantity.

Macerate the Powdered Opium for twenty-four hours in a wide-mouthed, well-closed bottle, with sufficient Purified Petroleum Benzin to completely cover it, shaking occasionally. Decant the liquid as closely as possible and repeat the treatment with Purified Petroleum Benzin. Again decant the liquid and pour the contents of the bottle into a plain filter contained in a glass funnel which should be well covered, drain, and then slowly percolate the residue with Purified Petroleum Benzin until the latter passes without color. Remove the filter containing the Opium from the funnel and expose the powder to the open air, so that it may dry thoroughly.

Deodorized Opium should be kept in well-stoppered bottles, and when assayed by the process given on p. 1025, should be found to yield not less than 12 per cent. nor more than 12.5 per cent. of crystallized morphine.

Opium in coarser powder may be deodorized in the same manner as directed above.

Remarks.—Deodorized (or *denarcotized*) *opium* is prepared by extracting powdered opium with benzin, that solvent removing little from the drug but its heavy narcotic odor and narcotine, an alkaloid to which many of the unpleasant after-effects of opium are said to be due. In the pharmacopœia of 1890 ether was used as the deodorizing solvent, but it has been found that benzin will do the work as thoroughly and more cheaply.

Medical Properties.—Similar to the powdered.

Dose.—65 milligrammes (1 grain).

CHAPTER LI

THE ALBUMINOIDS

THE albuminoids or proteins represent a group of complex organic substances, containing hydrogen, oxygen, nitrogen, and sulphur. The chemistry of these bodies is so complex that very little is known of their exact composition. Different proteins, however, possess different proportions of the elements above mentioned, and vary in constitution from simpler bodies, like protamine, to very complex substances, which can be split into many different chemicals.

All proteins seem based upon the urea molecule, to which is attached a number of other bodies. Among these bodies, constituting part of the protein molecule, may be mentioned nitrogenous bodies, like argenin and glycoll, sulphurated bodies, carbohydrate derivatives, like levulinic acid, phosphoric acid, etc.

The following classification of proteins is that adopted in 1907 by the Joint Committee of Chemical and Physiological Societies:

PROTAMINES.—Simplest form of albumin. Example, salmine ($C_{30}H_{57}N_{17}O_6$), found in salmon spawn.

HISTONES.—More complex bodies isolated from blood-corpuscles. Precipitated by ammonia.

ALBUMINS.—Soluble in water, diluted acids, alkalis, saturated solution of sodium chloride. Coagulated by heat. Examples, serum-albumin, egg-albumen, milk-albumin, and the albumin of seeds.

GLOBULINS.—Insoluble in water, soluble in dilute solution of sodium chloride. Coagulate on boiling. Examples, myosin (from lean meat), fibrinogen (the blood constituent that clots), serum globulin, and vegetable globulins.

SCLEROPROTEINS.—Like keratin and gelatin.

PHOSPHOPROTEINS, like vitellin (from yolk of egg) and caseinogen.

CONJUGATED PROTEINS.—

Nucleoproteins.—Such as are isolated from yeast-cells.

Glucoproteins.—Insoluble in water, soluble in 60 per cent. alcohol. Example, mucin.

Chromoproteins.—Like hemoglobin.

PROTEIN DERIVATIVES.—

Metaproteins.—Insoluble in water, soluble in acids and alkalis. Example, syntonin.

Proteoses.—Partly soluble in water. Products of digestion of albumin with vegetable enzymes like papain. Found in many seeds.

Peptones.—Perfectly soluble in water. Not precipitated by sodium chloride.

Polypeptides.—Mostly synthetic substances; some products of hydrolysis of proteins.

Serum-albumin is of particular interest to us inasmuch as, in the derangement of the human system producing the complaint called Bright's disease, the serum-albumin of the blood passes through the diseased kidney tissue and appears in the urine. The significance of the presence of albumin in the urine is beyond the limits of this book, and for further particulars on this interesting and important subject the reader is referred to standard works on urinary analysis.

Egg-albumen is of interest as an important food-stuff. While ordinary egg-albumen is difficult to crystallize, its crystallization has been accomplished by Hopkins. Egg-albumen combines with iron

salts to form iron albuminate, which dissolves in dilute alkali. The soluble (scale) form of this chemical is made with sodium citrate. A solution of it (*liquor ferri albuminati*) is found in the National Formulary.

Coagulated egg-albumen, digested with pepsin and acid, dissolves with the formation of *peptone*, which has the same percentage composition as pepsin. This is a basis of the United States Pharmacopœia test for the presence of pepsin (p. 934). Most commercial peptones are made from lean beef instead of from eggs. Peptone, like egg-albumen, forms a soluble iron salt—iron peptonate. *Solution of peptonate of iron*, containing $\frac{65}{100}$ of 1 per cent. metallic iron, is recognized by the National Formulary.

Similar are the peptomanganese preparations, a typical one of which is *liquor peptonati cum mangano*, N. F.

Phytoalbumin is of pharmaceutical interest merely because it makes up the larger bulk of horny seeds—an official example being the albumin of *nux vomica*.

Globulin is of passing interest because one form of it is a constituent of albuminous urine, and differs from serum-albumin by being precipitated by magnesium sulphate.

Fibrinogen is an albuminoid of the blood, the clotting of which is caused by the conversion of the substance into fibrin.

Vitellin is the albuminoid found in yolk of egg.

Syntonin is acid albumin, and can be made by boiling egg-albumen with nitric acid. It is here mentioned because its formation often misleads the tyro in urinary analysis in the nitric acid test. If nitric acid be added to urine containing albumin, the latter coagulates and precipitates. If too much acid is added, syntonin is formed, and no precipitation occurs.

Legumin is found in the seed of the Leguminosæ. It is soluble in diluted alkali.

Casein is the chief albuminoid of milk, from which it can be separated by means of rennet, acids, and some salts, such as the alums.

Nuclein is the albuminoid of the cell nucleus, that center of activity of the living cell.

Hemaglobin is an iron-bearing constituent of the blood, to which it transmits its red color. By treating defibrinated blood with acidulated pepsin and exactly neutralizing with sodium carbonate there is precipitated, *Hematin*, which has been recommended as a remedy for anemia, since it contains iron in easily assimilable form. Its crystalline hydrochloric acid compound is called *hemin*.

GELATINUM. Gelatin.

The purified air-dried product of the hydrolysis of certain animal tissues, as skin, ligaments, and bones, by treatment with boiling water.

An amorphous, more or less transparent solid, usually shredded or in thin sheets; colorless or with a slight yellowish tint, inodorous, and having a slight, characteristic, almost insipid taste. Unalterable in the air when dry, but putrefying rapidly when moist or in solution.

When incinerated, Gelatin is decomposed, leaving a slight mineral residue which should not exceed 2 per cent. of the original weight.

Gelatin is insoluble in cold water, but swells and softens when immersed in it, gradually absorbing from 5 to 10 times its weight of water. It is soluble in boiling

water, acetic acid, and glycerin; insoluble in alcohol, ether, chloroform, benzene, carbon disulphide, and fixed and volatile oils.

When dissolved in boiling water (1. in 50), it should solidify upon cooling, and form a transparent jelly.

An aqueous solution of Gelatin (1 in 5000) is at once rendered turbid on the addition of tannic acid T.S., the precipitate being insoluble in the presence of an excess of the reagent.

Gelatin is precipitated from its aqueous solution by mercuric chloride T.S. in excess; it is not precipitated by a solution of alum, by ferric chloride T.S., or by lead acetate T.S.

If potassium dichromate T.S. be added to a solution of Gelatin in hot water, the jelly which forms on cooling becomes insoluble in warm water after exposure to light.

Gelatin is prepared from bones by boiling same in water under pressure (as in a boiler), skimming the solution of gelatin to remove fat, and clarifying if necessary. On cooling the gelatin solidifies, and this is cut into cakes and dried on fish-nets. This explains the netted marking on the best quality of the sheet gelatin.

Glue is an impure gelatin made by digesting all kinds of gelatinous animal refuse—hide, horns, hoofs, as well as bones—by the process just given.

Gelatin is not soluble in cold water, but swells therein to form a jelly. It dissolves in boiling water, and is not precipitated from this solution by nitric acid nor by potassium ferrocyanide, as is egg-albumen. The precipitate formed when its solution is treated with tannin is similar to leather (p. 791). It will be noticed from the pharmacopœial tests that its jelly is made impervious to moisture by treatment with potassium dichromate. Formaldehyde acts in the same way, and these two chemicals are now used for tanning finer grades of leather.

GELATINUM GLYCERINATUM. Glycerinated Gelatin.

<i>Recipe.</i> —Gelatin, one hundred grammes.....	100 Gm.
Glycerin, one hundred grammes.....	100 Gm.
Water, a sufficient quantity,	
To make two hundred grammes..	200 Gm.

Pour upon the Gelatin sufficient Water, which has been previously boiled and cooled, to cover it; allow it to stand one hour; pour off the Water and allow the Gelatin to drain for a few minutes; then transfer it to a tared dish, add the Glycerin, and heat it on a water-bath until the Gelatin is dissolved. Strain the solution while hot, and continue to heat on the water-bath, until the product weighs *two hundred grammes*. When cold, cut the mass into pieces, and preserve these in suitable containers.

Remarks.—The faintly straw-colored transparent jelly thus produced is used as a suppository base (p. 334), although, from what has been said above concerning the formation of leather from gelatin, it is clear that this base should not be used in making tannin suppositories. It is important that all the water be driven off in making the mass, as provided by the pharmacopœia, for if any remains, the mass grows mouldy and turns dark brown.

Keratin is the chief constituent of horny tissue, and is obtained therefrom by digestion in artificial gastric juice (diluted hydrochloric acid plus pepsin) until all albuminous matter is dissolved. The

washed insoluble residue is then macerated several weeks with ammonia, and the ammoniated liquid on evaporation yields keratin as a sticky mass.

It is insoluble in acids, but soluble in alkali, hence is of medicinal value as a coating for enteric pills (p. 321).

PEPSINUM. Pepsin.

A proteolytic ferment or enzyme, obtained from the glandular layer of the fresh stomach of the hog (*Sus scrofa*, var. *domesticus* Gray), and proved to be capable, when assayed by the process given below, of digesting not less than 3000 times its own weight of freshly coagulated and disintegrated egg-albumen.

If it is desired to use a diluent for reducing Pepsin of a higher digestive power to that required by the Pharmacopœia, sugar of milk should be employed for this purpose.

Lustrous white, pale yellow or yellowish, transparent or translucent scales or grains, or a fine white cream-colored amorphous powder, free from any offensive odor, and having a slightly acid or saline taste. It should be not more than slightly hygroscopic.

Soluble or almost entirely soluble in about 50 parts of water, the solution having more or less opalescence; more soluble in water acidulated with hydrochloric acid; insoluble in alcohol, ether, or chloroform.

Pepsin, when in solution, is incompatible with alkalis, alkaline earths, or alkali carbonates. The presence of hydrochloric acid of greater strength than 0.5 per cent. inhibits and rapidly destroys its proteolytic activity. Its solution is precipitated by the salts of many heavy metals, and by tannic acid or gallic acid. Pepsin and pancreatin in solution are incompatible with one another; if the solution be neutral or alkaline, the pancreatin gradually destroys the Pepsin, and if acid, the Pepsin destroys the pancreatin.

On heating a solution of Pepsin in acidulated water to 100° C. (212° F.) it becomes milky, or yields a light flocculent precipitate, and loses all proteolytic power; in a dry state it is not injured if subjected to the above temperature. The activity of Pepsin in solution is destroyed by temperatures exceeding 70° C. (158° F.). Pepsin usually has a slightly acid reaction; it may be neutral, but should never be alkaline.

ASSAY OF PEPSIN

Pepsin, one-tenth gramme.....	0.1 Gm.
Egg Albumin, boiled and disintegrated, ten grammes.....	10.0 Gm.
Diluted Hydrochloric Acid,	
Distilled Water, each, a sufficient quantity.	

Mix 9 Cc. of diluted hydrochloric acid with 291 Cc. of distilled water, and dissolve the Pepsin in 150 Cc. of the acid liquid. Immerse a hen's egg, which should be fresh, during fifteen minutes in boiling water; remove the pellicle and all of the yolk; rub the white, coagulated albumin through a clean No. 40 sieve. Reject the first portion that passes through the sieve, and place 10 Gm. of the succeeding portion in a wide-mouthed bottle of 100-Cc. capacity. Add 20 Cc. of the acid liquid, and with the aid of a glass rod tipped with cork or black rubber tubing, completely disintegrate the albumin; then rinse the rod with 15 Cc. more of the acid liquid and add 5 Cc. of the solution of Pepsin. Cork the bottle securely, invert it three times, and place it in a water-bath that has previously been regulated to maintain a temperature of 52° C. (125.6° F.). Keep it at this temperature for two and one-half hours, agitating every ten minutes by inverting the bottle once. Then remove it from the water-bath, add 50 Cc. of cold distilled water, transfer the mixture to a narrow graduated cylinder, and allow it to stand for half an hour. The deposit of undissolved albumin should not then measure more than 1 Cc.

The relative proteolytic power of Pepsin stronger or weaker than that just described may be determined by ascertaining through repeated trials the quantity of the above Pepsin solution required to digest, under the prescribed conditions, 10 Gm. of boiled and disintegrated egg-albumen. Divide 15,000 by this quantity expressed in Cc. to ascertain how many parts of egg-albumen one part of the Pepsin will digest.

Tests for Identity.—Given above.

Assay.—Given above.

Extraction.—The mucous membrane of the hog's stomach is treated with water acidulated with hydrochloric acid at a temperature of 38° to 45° C. until the entire mass is fluid, chloroform or sulphurous acid being used to prevent putrefaction, and the liquid allowed to stand over night, whereupon the fluid portion is strained from the sediment, evaporated in vacuo to the consistence of a syrup, which is scaled on plates of glass.

Composition.—While absolutely pure pepsin is not known, it is supposed to be protein compound. The commercial pepsin, even of the highest strength, contains foreign matter, such as mucus, peptones, and syntonin. It will be seen from the official description that the test for pepsin is its ability for dissolving egg-albumen, one grain of the official pepsin being supposed to have the power of dissolving 3000 grains of hard-boiled white of egg when digested at the proper temperature in an acid mixture representing very closely the acidity of the stomach. Such pepsin is designated as 1 : 3000. Other pepsins of weaker strength are known (such as 1 : 1000 and 1 : 500), in each case the larger number representing the number of grains of albumin which one grain of the pepsin can dissolve. These weaker pepsins can be used in place of the 1 : 3000 pepsin, provided a proportionally larger quantity is employed, the proportion needed being inversely the amount of egg-albumen which their pepsin dissolves. Thus, if we used 1 : 1800 pepsin in place of 1 : 3000 pepsin, for every 1800 grains of pepsin of 1 : 3000 strength, we will require 3000 grains of the 1 : 1800. It can be seen from this statement that such problems are solved by alligation, the method being exactly identical with the reduction of alcohol of a certain strength to that of another strength.

Pepsin is a valuable aid to digestion. It is administered as compound digestive powder, N. F.; essence of pepsin, N. F.; glycerite of pepsin, N. F.; elixir of pepsin, N. F., and compound digestive elixir, N. F.

Of these preparations, the glycerite is the only one that is effective, as the alcohol of the essence and elixir and the alkali and pancreatin of the compound digestive powder and compound digestive elixir make the pepsin inert.

Dose.—250 milligrammes (4 grains).

PANCREATINUM. Pancreatin.

A mixture of the enzymes naturally existing in the pancreas of warm-blooded animals, usually obtained from the fresh pancreas of the hog (*Sus scrofa*, var. *domesticus* Gray) or the ox (*Bos taurus* Linné), and consisting principally of amylopsin, myopsin, trypsin, and steapsin, and proved to be capable, when assayed by the process given below, of converting not less than 25 times its own weight of starch into substances soluble in water.

A cream-colored, amorphous powder, having a faint, peculiar, not unpleasant odor, and a somewhat meat-like taste.

Slowly soluble in water and containing not more than 10 per cent. of substances insoluble in this solvent; insoluble in alcohol.

Pancreatin digests albuminoids and converts starch into sugar, dextrin, or maltose; it exhibits its peculiar activities in neutral, faintly alkaline, and faintly acid media; more than traces of mineral acids or large amounts of alkalis render it inert.

Alkali carbonates exert slightly inhibitory power upon Pancreatin. The digestive power of Pancreatin is injured by contact with pepsin in solution.

If 0.28 Gm. of Pancreatin and 1.5 Gm. of sodium bicarbonate be added to 100 Cc. of tepid water contained in a flask, and if 400 Cc. of fresh cows' milk, which has been previously heated to 38° C. (100.4° F.), be then added, and the temperature of the mixture maintained at this point for thirty minutes, the milk should be so completely peptonized that, if a small portion of it be transferred to a test-tube and mixed with some nitric acid, no coagulation should occur.

ASSAY OF PANCREATIN

Pancreatin, *three-tenths of a gramme*..... 0.3 Gm.
 Starch, dry and in fine powder, *seven and one-half*
grammes..... 7.5 Gm.
 Distilled Water,
 Tenth-normal Iodine V.S., each, *a sufficient quantity*.

Introduce the starch into a flask, add 120 Cc. of distilled water, and boil until a translucent mixture results. Cool the resulting paste to 40.5° C. (105° F.), and add to it the Pancreatin, previously dissolved in about 10 Cc. of distilled water at 40.5° C. (105° F.). Shake the flask well, maintaining the temperature of the mixture at 40.5° C. (105° F.) during five minutes; at the end of this time all of the starch should be converted into substances soluble in water, and a thin liquid be produced. Mix 2 drops of tenth-normal iodine V.S. with 60 Cc. of distilled water, and add to it 2 drops of the warm converted starch solution; no color should result, or, at most, a wine-red color, showing the presence of dextrin and maltose. The appearance of a blue or purple color indicates the presence of *unconverted starch* and that the Pancreatin is below the standard—*i. e.*, that of converting not less than 25 times its own weight of starch into substances soluble in water.

Tests for Identity.—Given above.

Assay.—Given above.

Composition.—Amylopsin, myopsin, trypsin, and steapsin (all albuminoids). Trypsin and myopsin digest albumin, amylopsin hydrolyzes starch, while steapsin emulsifies fats.

Extraction.—The minced pancreas is macerated for some time with water containing enough chloroform to prevent putrefaction. The liquid is then expressed from the residue, is filtered, and rapidly evaporated at a temperature not exceeding 45° C.

Remarks.—The test for pancreatin is the estimation of its peptonizing power; that is, 0.28 gramme of pancreatin and 1.5 grammes of sodium bicarbonate should be capable of converting the albumin of 400 Cc. cows' milk into peptone, which is not coagulated by nitric acid. Not only is this process used as a test, but the *peptonized milk* itself is sometimes used as a diet where the stomach cannot tolerate the natural albumin of milk.

The assay of pancreatin is based on its ability to convert starch into dextrin and maltose; the requirements being that one part of pancreatin should be able to transform at least 25 times its weight of starch.

While pepsin acts as in acid medium, pancreatin is of value only when combined with mild alkalis, and this emphasizes the absurdity of prescribing these two substances in combination, a medium which will develop the activity of pepsin rendering pancreatin inert.

Pepsin and pancreatin are of value in dyspepsia, both acting as food digestants; their natural function in the animal economy. The two differ, however, as to the kind of food each digests, pepsin digesting albuminoid matter of the stomach, while pancreatin digests albumin-

THE ALBUMINOIDS

oids, starch, and fat in the intestines. To be effective, pe should be administered only in enteric pills. It is a consti peptonizing powder, N. F., of compound powder of pepsin, N. of compound digestive elixir, N. F.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

Diastase is the ferment (or enzyme) found in malt, from which it is obtained by maceration of malt with cold water, and then pouring the filtered infusion into double the amount of strong alcohol. Taka diastase is prepared by treating wheat bran with a peculiar fungus, called in Japan "moyashi," extracting the diastase thus formed with water, and precipitating with alcohol. Diastase converts starch into dextrin and maltose, and is, therefore, used in starchy indigestion.

Dose.—300 milligrammes (5 grains).

Papain is the ferment obtained from the milk juice of *Carica papaya*, and has been recommended as a general digestant and for dissolving the diphtheritic membrane. Do not confuse this with a liquid nostrum, *papaine*, which is used as a sedative.

Emulsin and *myrosin* are ferments found respectively in bitter almond and black mustard, and have already been considered under those headings. Of their chemistry but little is known.

Flesh is a combination of fibrous tissue with blood, albuminoids, and a host of other substances, such as the urea derivatives, sarcin and carnin.

Meat preparations used for invalid diet are a part of the stock of the modern drug-store, hence a few words concerning these are in place. Of these meat products the only ones of real nutritive value are *meat-juices*, made by expressing the juice from a slightly broiled steak. These juices decompose as rapidly as does meat itself, hence must be either fortified by preservatives or dispensed in a perfectly fresh condition. The writer knows of several progressive pharmacists who have built up a large trade in meat-juice, preparing same fresh every day.

Beef-tea is made by digesting finely chopped meat with warm water and straining.

Beef-extracts represent beef-tea evaporated in vacuo—being administered redissolved in warm water. Both of these products are practically devoid of nutritive qualities, its stimulating action being due to the sarcin and carnin they contain, these bodies acting similarly to their allied body, caffeine. One authority has claimed that "beef-tea" has no more nutritive action than has urine.

Milk is caseine emulsion of fat (butter), containing milk-sugar and inorganic salts.

When milk is treated with yeast, the milk-sugar ferments to lactic acid, and if the operation is carefully conducted, we obtain the pharmaceutical called *koumiss*. A recipe for this liquor is found in the National Formulary under the name of "*lac fermentatum*."

The original koumiss was made in Russia by fermenting mares' milk with a peculiar ferment, kephir.

Condensed milk is milk evaporated in vacuo, and usually preseved by addition of cane-sugar.

Solid evaporated milk is now on the market; one brand—"Nutrium"—consists of 40 per cent. proteids, 45.7 per cent. sugar of milk, 8.57

per cent. lime salts and other minerals, and 5.73 moisture. It is said that 100 pounds skim milk give 10 pounds nutrium.

Infant's food, of which there are a great host, can be grouped into the following classes:

Milk Foods.—Such as condensed milk. Bad as exclusive infant diet.

Predigested Foods.—Milk foods digested with pepsin or pancreatin.

Cereal Foods.—Grains dextrinized by heat. To be used mixed with milk.

Malted Foods.—Cereals digested with milk and then dried. Used mixed with milk.

Mixed Foods.—(a) Combinations of extract of barley or wheat with powdered predigested milk.

(b) Dextrinized cereals with unpredigested milk powder.

ANIMAL DRUGS

At this place we will discuss those drugs of animal origin—whole animals, animal organs, and animal excretions—which are of such complex composition that they cannot be classed among definite chemicals. As examples of animal products that are definite chemicals may be cited *lactic acid* (p. 677), the animal *fats, oils, and waxes* discussed in Chapter XLI., and the animal proteins, *pepsin and pancreatin*, discussed on the foregoing pages of this chapter. The discussion of the other animal products in this chapter may be misleading, inasmuch as some of the products here considered are not albuminoidal in character. However, as the characteristic compounds of most living animal tissue are protein matter, it seems appropriate to discuss animal drugs at this place, with the warning given above.

The drugs to be considered may be classified as follows:

Whole Animals.—Cantharides, U. S. P., and Cochineal, U. S. P.

Animal Organs.—Egg (or the yolk, Vitellus, U. S. P. 1890), isin-glass, U. S. P. 1890, the musk-sac (the contents thereof is official), thyroïdal glands, U. S. P., suprarenal glands, U. S. P.

Animal Excretions.—Oxgall, U. S. P.; milk (p. 678).

Antitoxins.—Antidiphtheritic serum, U. S. P.; other serums. Vaccine Virus.

CANTHARIS. Cantharides. Spanish Fly.

The beetle, *Cantharis vesicatoria* (Linné) De Geer, thoroughly dried at a temperature not exceeding 40° C. (104° F.).

Constituents.—*Cantharidin*, gum, fixed oil, etc. Cantharidin, the vesicating principle, has the composition $C_{10}H_{12}O_4$, and is the anhydride of cantharidinic acid, $C_{10}H_{12}O_3(OH)_2$.

Remarks.—Since cantharidin is a distinctive neutral principle, this drug might be discussed more properly in Chapter XLVIII.

Cantharides are beetles, which differ from flies in having cases into which they fold their wings. The active principle is cantharidin, which produces the irritating effects of the drug, and which is sublimable. Hence care must be observed in hovering over melting cantharides cerate, as thereby severe irritation of the eyes may ensue.

Medical Properties.—Aphrodisiac, vesicant, stimulant to scalp. Administered internally (rarely nowadays) in the form of the official *tincture* (p. 224). Externally as the official *cerate* (p. 341) properly spread (fly blister).

Dose.—Of drug, 30 milligrammes ($\frac{1}{2}$ grain).

COCCUS. Cochineal.

The dried female insect, *Pseudococcus cacti* (Linné) Burmeister.

Constituents.—*Carminic acid*, wax, and fat; not more than 6 per cent. ash.

Remarks.—Cochineal is the female insect gathered while with young. Like cantharides it might more properly be considered in Chapter XLVIII., as carminic acid is grouped among the class of proximate principles called natural colors. This body, $C_{11}H_{12}O_7$, has been proved to be a derivative of naphthoquinone (p. 866). *Carmine* is the insoluble form of carminic acid, made by precipitating an infusion of cochineal with either alum or potassium bitartrate. The ash requirement given above is based on the fact that cochineal is largely adulterated with mineral matter.

Medical Properties.—Used entirely as a color, the 10 per cent. tincture (menstruum, 80 per cent. alcohol) being employed. For this purpose, however, solution of carmine, N. F., is preferable.

Vitellus, U. S. P. 1890, is the yolk of the egg. It contains 30 per cent. fixed oil, emulsionized by the proteid *vitellin*, and is used as a protective dressing in burns and for making emulsion of cod liver oil (p. 267).

Ichthyocolla, U. S. P. 1890, or *isinglass*, represents the dried "sounds" (swimming bladders) of the Russian sturgeon, *Acipenser Huso*, whose eggs (roe) constitute that delicacy, caviar.

It is almost pure gelatin, and is the adhesive constituent of court plaster. (*Emplastrum Ichthyocollæ*, U. S. P. 1890.)

MOSCHUS. Musk.

The dried secretion from the preputial follicles of *Moschus moschiferus* Linné.

Constituents.—Ammonia, cholesterin (p. 941), fat, wax, odorous principle.

Remarks.—By "preputial follicles" is meant a little sac situated between the navel and the penis of the musk deer (*Moschus moschiferus*), an animal that lives in the mountains of Central Asia. Official musk is the "secretion"—the contents—of this sac. It is used largely in perfumery. Bear in mind that artificial musk (p. 815) has nothing in common with the drug, save its similar perfume.

Medical Properties.—Powerful stimulant, of especial value in the exhaustion that comes with pneumonia. While little used, it is badly needed when desired, and that of the best quality; hence the pharmacist should always be ready to furnish a high grade of musk on demand. The tincture (p. 231) is official.

Dose.—250 milligrammes (4 grains).

GLANDULÆ THYROIDEÆ SICCÆ. Desiccated Thyroid Glands.

The thyroid glands of the sheep (*Ovis aries* Linné), freed from fat, and cleaned, dried, and powdered.

A yellowish, amorphous powder, having a slight, peculiar odor, and containing the active ingredient of the thyroid tissue; partially soluble in water.

One part of Desiccated Thyroid Glands represents approximately 5 parts of the fresh glands.

Upon incineration it should yield not more than 6 per cent. of ash.

If 1 Gm. of Desiccated Thyroid Glands be mixed with an equal weight of pure sodium hydroxide and carefully fused in a silver dish, and oxidized by adding potassium nitrate while fusing, until a white mass remains, and if the fused mass be dissolved in a small quantity of water, the solution treated with 2 Gm. of sodium nitrite, acidified with concentrated nitric acid, and then shaken with 5 Cc. of chloroform, a decided pink to violet coloration should be imparted to the latter (presence of *iodine compounds*). A cold extract of Desiccated Thyroid Glands, treated with 2 Gm. of sodium nitrite and acidified with strong nitric acid, should not give the iodine test on shaking with chloroform.

Constituents.—Thyroidin, combined with proteid matter, etc. The “thyroid gland” is a small sac found in the neck. Its abnormal development forms the disease called goiter.

Remarks.—Thyroidin owes its activity largely to its iodine content, and some specimens contain as much as 14 per cent. of that element. The last test given above shows—first, that iodine is present, and, secondly, that it is present in organic combination.

Medical Properties.—Furnishes iodine in non-irritating and easily absorbable form. Used in goiter, obesity, etc.

Dose.—250 milligrammes (4 grains).

GLANDULÆ SUPRARENALIS SICCÆ. Desiccated Suprarenal Glands.

The suprarenal glands of the sheep (*Ovis aries* Linné) or ox (*Bos taurus* Linné), freed from fat, and cleaned, dried, and powdered.

A light yellowish-brown, amorphous powder, having a slight, characteristic odor; partially soluble in water.

One part of Desiccated Suprarenal Glands represents approximately 6 parts of fresh glands, free from fat.

Upon incineration it should not yield more than 7 per cent. of ash.

If 0.5 Gm. of Desiccated Suprarenal Glands be macerated with 25 Cc. of water for fifteen minutes, and filtered, the filtrate should give an emerald-green color upon the addition of a few drops of ferric chloride T.S. The green color disappears quite rapidly.

Constituents.—Epinephrin, fat, proteids, ash, etc.

Remarks.—The word “suprarenal” means “above the kidney,” and explains the location of this gland in the animal body. Epinephrin is a basic body described on p. 795.

This alkaloid in a more or less pure state is now largely advertised under the names *adrenalin*, *suprarenin*, *adrin*, etc.

Medical Properties.—Increases blood-pressure, hence of value in shock. Relieves inflammation of mucous membrane, hence recommended for catarrh, hay-fever, etc.

Dose.—Of glands, 250 milligrammes (4 grains). For external use an aqueous or oily solution of the active principle (1 : 100,000) is preferable.

FEL BOVIS. Oxgall.

The fresh bile of *Bos taurus* Linné.

A brownish-green or dark green, somewhat viscid liquid, having a peculiar, unpleasant odor, and a disagreeable, bitter taste.

Specific gravity: 1.015 to 1.025 at 25° C. (77° F.).

It is neutral or has a faintly alkaline reaction upon litmus-paper.

A mixture of 2 drops of Oxgall and 10 Cc. of water, when treated, first, with a drop of a freshly prepared solution of 1 part of sugar in 4 parts of water, and afterward with sulphuric acid, cautiously added, until the precipitate first formed is redissolved, gradually acquires a brownish-red color, changing, successively, to carmine, purple, and violet.

Constituents.—Sodium salts of *glycocholic acid* and *taurocholic acid*; *lecithin*; *cholesterin*; the bile-pigments, bilirubin, biliverdin, etc.; mucin; water, 85 per cent. The two acids above mentioned are combinations of *cholalic acid*, $C_{24}H_{40}O_5$, with glycocoll (p. 673), and with taurine (p. 673) respectively. Cholesterin is an alcohol ($C_{24}H_{45}OH$) whose chemistry is little understood. *Isocholesterin* will be remembered as the alcohol with which oleic, stearic, and palmitic acids are united in wool-fat (p. 734).

Remarks.—Bile is the secretion of the liver, and is designed to emulsionize and assimilate fats. The test given above, depending on the play of colors, when bile is treated with sugar and sulphuric acid (Pettenkofer's test), is very characteristic.

Medical Properties.—Laxative and cholagogue. Almost invariably used in the purified form.

FEL BOVIS PURIFICATUM. Purified Oxgall.

Recipe.—Oxgall, three hundred cubic centimeters..... 300 Cc.

Alcohol, one hundred cubic centimeters..... 100 Cc.

Evaporate the Oxgall, in a tared porcelain dish, on a water-bath, to about one hundred grammes, then add to it the Alcohol, mix the whole thoroughly, and set it aside, well covered, for three or four days. Then decant the clear solution, filter the remainder, and, having mixed the liquids and distilled off the alcohol, evaporate the residue to a pilular consistence.

A yellowish-green, soft solid, having a peculiar odor, and a partly sweet and partly bitter taste.

Very soluble in water and in alcohol.

A solution of 1 part of Purified Oxgall in about 100 parts of water behaves toward sugar and sulphuric acid in the same manner as the solution mentioned under *Fel Bovis*.

An aqueous solution of Purified Oxgall should be clear, and should remain transparent upon the addition of an equal volume of alcohol (evidence of proper purification).

Remarks.—This consists of oxgall dissolved in alcohol to remove the mucin and other mucilaginous bodies, and after distilling off the alcohol, evaporating the residue to pilular consistence. It is an improved form of the old-fashioned inspissated oxgall.

Medical Properties.—Like oxgall.

Dose.—500 milligrammes ($7\frac{1}{2}$ grains).

SERUM ANTIDIPHThERICUM. Antidiphtheric Serum.
Diphtheria Antitoxin.

A fluid separated from the coagulated blood of a horse (*Equus caballus* Linné), immunized through the inoculation of diphtheric toxin. It should be kept in sealed

glass containers, in a dark place, at temperatures between 4.5° and 15° C. (40° and 59° F.).

A yellowish or yellowish-brown, transparent or slightly turbid liquid, odorless or having a slight odor due to the presence of the antiseptic used as a preservative.

Specific gravity: 1.025 to 1.040 at 25° C. (77° F.).

Antidiphtheric Serum gradually loses its power, the loss in one year varying between 10 and 30 per cent. Each container should be furnished with a label or statement, giving the strength of the Antidiphtheric Serum, expressed in antitoxic units, the name and percentage by volume of the antiseptic used for the preservation of the liquid (if such be used), the date when the Antidiphtheric Serum was last tested, and the date beyond which it will not have the strength indicated on the label or statement.

The standard of strength, expressed in units of antitoxic power, should be that approved or established by the United States Public Health and Marine Hospital Service.

Remarks.—The subject of antitoxins, of which this new official is the most prominent example, is more medical than pharmaceutical, and these products can never be made by the retail pharmacist, a large well-equipped and aseptic plant being essential to their successful manufacture. This fact caused the question of the admission of these products into the pharmacopœia, to become the most widely discussed problem of the last revision, and the final decision to admit the antidiphtheric serum was based on the desire to secure a uniform standard for this important commodity.

As to the theory of antitoxins, Behring showed that most animals when infected with certain diseases are capable of generating in their blood-serum substances (antitoxins) capable of neutralizing the poisonous matter (toxins) transmitted to the blood by the organism producing the disease; and, further, that the question as to whether the animal survives or succumbs to the disease is a question as to which predominates, the toxin or the antitoxin.

Applying this idea in the manufacture of diphtheria antitoxin, the animal (usually a horse) is inoculated with cultures of diphtheria germs, first in small doses then in large ones. A mild case of the disease is thus produced in the animal, which immediately combats the toxin by the formation of the antitoxin. As increased doses of the toxin are administered, an increased amount of antitoxin is produced until, at the end of two weeks, the animal has stored up so much antitoxin in its blood-serum that it becomes "immune."

A certain quantity of blood (5 to 10 liters) is then removed through the jugular vein, allowed to stand until the albuminoids separate by coagulation, and the liquid portion (the serum) is then mixed with some antiseptic (phenol, thymol, etc.), is standardized, and then put into glass tubes which are immediately sealed.

The pharmacopœia directs that the standard for antitoxin—"immunity unit"—be that of the United States Public Health and Marine Hospital Service; namely, the times that quantity of antitoxin which will completely protect a 250-gramme guinea-pig against 100 times the least fatal dose of the corresponding toxin. A brief explanation of this statement is in order.

The toxin (as the culture of the germ is called) is prepared and administered to, say, a dozen guinea-pigs of the same weight (250 grammes); to each a different dose. The smallest amount killing one of the guinea-pigs within four days is "the minimum fatal dose."

One hundred times this quality of toxin is mixed with various amounts of the antitoxin serum, and administered to a second batch of guinea-pigs. The smallest amount of the antitoxic serum which prevents the hundred-fold dose of toxin from being fatal to the guinea-pig represents the "immunity unit"; 100 times this volume of serum represents 100 units; 3000 times this volume represents 3000 units, and so on.

The amounts of toxin and antitoxin serum involved in guinea-pig testing is very small— $\frac{1}{200}$ Cc. of the toxin is apt to be fatal; while $\frac{1}{300}$ Cc. of the antitoxic serum often represents one "immunity unit." Hence the tests are carried out with diluted toxin and antitoxin of known strength.

The absolute strength of the same volume of different batches of serum may vary materially, hence the dosage is by "immunity units" and not by volume. Thus in one case 3000 units may be in 30 Cc. of serum; in another in 50 Cc. of serum.

This is but an outline of the method of standardization, and for further details the reader is referred to a paper by C. T. McClintock (Proceedings A. Ph. A. 44, p. 254).

Medical Properties.—Curé of and immunization against diphtheria.

Dose.—3000 units. Immunizing dose for well persons, 500 units. Always by hypodermic injection.

Similar *antitoxins* for *tetanus*, *snake-bite*, *bubonic plague*, and *glanders* are now on the market, and all prepared by a similar process, namely, from the serum of animals infected with the special disease for which the antitoxin is intended.

Vaccine virus is the virus from the sores upon a heifer which has been inoculated with cowpox. While antitoxins contain the principles protecting one against the poison of a disease, vaccines contain the substance that produce the disease. Thus vaccine virus gives a man the comparatively mild disease, cowpox, and the sufferer develops in his blood the cowpox antitoxin, which protects him from the ravages of small-pox.

The virus was formerly marketed as a dry coating on ivory points or on quills. Now it is almost always dispensed in glycerinic solution in sealed tubes.

Note that vaccine virus, as well as the antitoxins, must be kept in a cool place, and that they deteriorate with age.

Opsonins represent the latest development of bacterial therapy. These are substances which, introduced into the blood, act on disease bacteria, so changing them as to cause them to be readily absorbed by the phagocytes, those bodies found in the blood, and upon whose preservation depends the well-being of the animal. Bacteria not so treated attack the phagocytes, producing the various bacterial diseases. Each species of bacteria has its special opsonin, and these opsonins, or bacterial vaccines, can be prepared by making cultures of the special bacteria, killing them, suspending in normal saline solution, and sterilizing at 60° C. For further details the reader is referred to a paper by Dr. L. H. Warner, *American Druggist*, December 1907, p. 389.

PART V
PHARMACEUTIC TESTING
CHAPTER LII.
INTRODUCTORY

ON the foregoing pages enough has been said to emphasize the importance of examining pharmaceuticals and chemicals, both as to composition and also the quantity of each component. The subject of such testing is called *analytic chemistry*, a branch of science the many details of which are clearly beyond the scope of this work. In the few chapters that follow only an outline of the principles of pharmaceutical testing as directed in the pharmacopœia will be given, and for complete details the reader is referred to one of the several books on analytic chemistry mentioned in the Preface.

Analytic chemistry is divided into two subdivisions—*qualitative analysis*, which aims to find the constituents of a compound, and *quantitative analysis*, by means of which we can estimate the quantity of each ingredient. Quantitative analysis is, in turn, divided into three classes: Gravimetric, volumetric, and gasometric analysis.

Gravimetric analysis is where the quantity of ingredient is estimated by conversion into an insoluble compound containing a definite proportion of the ingredient, collecting and weighing the precipitate, and from the weight obtained estimating the amount of the ingredient by molecular proportions.

Volumetric analysis (or *titration*) is where the quantity of ingredient is estimated from the amount of a definite standard solution of a chemical (*volumetric solution*) required to combine with it exactly; the amount of this volumetric solution required exactly to complete the reaction is usually denoted by the change in color produced by a third chemical, called an *indicator*.

Assaying is a term applied in pharmacy to the quantitative estimation of the active ingredient of a mixed product, notably a drug. As the quantity is sometimes estimated by gravimetric analysis and sometimes by volumetric analysis, the process is merely a modification of these, the only difference being that some process of extraction must first be employed.

The *apparatus* used in pharmaceutical testing have been referred to in the chapters comprising Part I. For qualitative analysis, funnels, beakers, spritz-bottles, flasks, evaporating dishes, filter-paper, and Bunsen burners are required, and these have already been described. In addition thereto it is necessary to have test-tubes and test-tube racks (p. 141); red and blue litmus-paper (which is filter-paper dipped in a decoction of the lichen, litmus, and treated with acid to make it red, or alkali to make it blue); blue glass; and platinum wire and foil.

The chemical to be analyzed is usually made into a solution and treated with solutions of other chemicals. Such solutions are called *test solutions*. This phrase is frequently abbreviated to T.S. (example, potassium iodide T.S.).

The test solutions are usually prepared by making a 10 to 20 per cent. solution of the chemical (called *reagent*) in distilled water, but many of the test solutions must be prepared by special treatment. A recipe for each test solution directed by the pharmacopœia is found in the appendix to that standard (pp. 517-568), to which the reader is referred.

These test solutions should be kept in special bottles, closed either with glass or rubber stoppers. Such a test solution bottle is shown in Fig. 228.

While most qualitative analyses are performed with test solutions, the reagent, hydrogen sulphide, is employed as a gas and is usually



FIG. 228.—Reagent bottle.

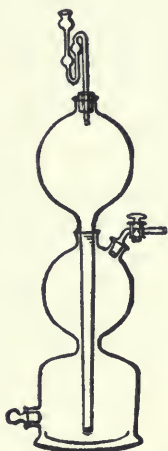


FIG. 229.—Kipp's gas generator.

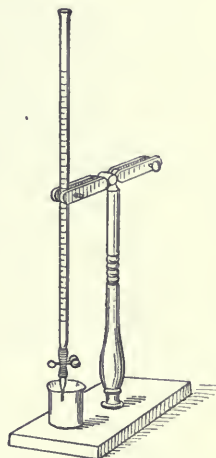


FIG. 230.—Burette and holder.

generated by action of sulphuric acid on ferrous sulphide in a Kipp's generator (Fig. 229), which acts automatically. Then sometimes the reagent is employed in solid form. Thus, borax is fused for the borax bead test, sodium carbonate and potassium nitrate are melted on platinum foil in several of the fusion tests, etc.

In quantitative analysis the apparatus used are crucibles, desiccators, analytic balances, measuring vessels, such as burettes (Fig. 230), pipettes, measuring cylinders, 100-Cc. and 1000-Cc. bottles. The reagents used are practically the same as in qualitative analysis, with the additional line of volumetric solutions, the manufacture of which will be discussed a few pages farther on.

In drug assays the apparatus is practically the same as that just mentioned, but in addition thereto, since the active principle is usually extracted by shaking out of the watery solution with ether or chloroform (separation from an immiscible solvent), a separatory funnel is required.

The detection of the ingredients of a compound in qualitative analysis is accomplished in several ways:

I. *By production of a characteristic precipitate*: thus a solution of a sulphate when mixed with barium chloride T.S. gives a white precipitate, insoluble in boiling nitric acid.

II. *By production of a characteristic color in a solution*: thus a diluted solution of copper sulphate gives a deep azure blue solution with excess of ammonia water.

III. *By production of a characteristic odor*: thus an acetate warmed with alcohol and sulphuric acid gives the pleasant odor of acetic ether.

IV. *By transmitting a characteristic color to a colorless Bunsen flame*: thus a trace of sodium salt on a loop of platinum wire placed in a Bunsen flame makes it a vivid yellow.

V. *By giving a characteristic color to a borax bead*: thus if borax is fused on a loop of platinum wire and a trace of a manganese salt added to the bead, which is then fused once more, the cooled solidified bead will be found to possess a beautiful amethyst color.

VI. *By giving a characteristic color to specially prepared test-paper*: thus borax gives buff colored turmeric paper a red-brown tint.

VII. *By producing a play of colors when brought in contact with the reagent on a porcelain surface*: thus most alkaloids give characteristic color reactions when treated with a drop of sulphuric acid and a trace of potassium dichromate.

VIII. *By leaving a characteristic residue on heating on charcoal with a blow-pipe*: thus zinc salts when treated with cobaltous nitrate and heated with a blow-pipe leave a vivid green spot.

It will be noticed that the pharmacopœia makes use of all of these several means of identity, and a classification of the qualitative tests of the pharmacopœia will comprise the next chapter.

CHAPTER LIII

THE QUALITATIVE INORGANIC TESTS OF THE PHARMACOPOEIA

Free Chlorine Liberates Iodine from Potassium Iodide

THIS test is used to detect free chlorine and free bromine in hydrochloric acid, U. S. P.

Sought as impurity in—

Hydrochloric Acid.—If 1 Cc. of the Acid be diluted with 5 Cc. of water, and if 1 Cc. of potassium iodide T.S. with 1 Cc. of chloroform be added, and the mixture agitated, the chloroform should be free from any violet coloration (absence of free chlorine or bromine).

Benzoic Acid.—See p. 779.

Hydrochloric acid and chlorides give, with silver nitrate T.S., a white curdy precipitate insoluble in nitric acid, but soluble in ammonia water. This precipitate is silver chloride (AgCl).

The following official chemicals are tested by this process:

PRESENT AS INGREDIENTS:

Hydrochloric Acid.—See p. 411.

Sodium Chloride.—As with *hydrochloric acid*.

Ammonium Chloride.—As with *hydrochloric acid*, but insolubility in nitric acid is omitted.

Calcium Chloride.—

Zinc Chloride.—

Ferric Chloride.—

Solution of Ferric Chloride.—

Tincture of Ferric Chloride.—

Corrosive Mercuric Chloride.—

As with *hydrochloric acid*, but solubility in ammonia water is omitted.

Ammoniated Mercury.—A solution of the compound in diluted nitric acid gives white precipitate with silver nitrate T.S.

Pilocarpine Hydrochloride.—Silver nitrate T.S. produces, in an aqueous solution of the salt, a white precipitate insoluble in nitric acid.

Quinine Hydrochloride.—Like test for *pilocarpine hydrochloride* just given.

Cocaine Hydrochloride.—Like test for *pilocarpine hydrochloride* just given, except strength of aqueous solution (1:100) given.

Chlorides Sought as Impurity.—The diversified modifications of the test necessary to adapt it to the different conditions found in the several chemicals make classification difficult, but the following chart gives a general outline. For details the reader is referred to the pharmacopœia itself.

CHEMICAL.	STRENGTH OR CHARACTER OF SOLUTION.	NITRIC ACID ADDED?	AMOUNT SILVER NITRATE T.S. USED.	RESULT.
<i>Boric acid</i> .	Aqueous solution, 1:25.	Yes.	No mention.	No precipitate.
<i>Nitric acid</i> .	Aqueous solution, 1:10.	None.	" "	" "
<i>Sulphuric acid</i> .	Aqueous solution, 1:20.	No.	" "	" "
<i>Zinc acetate</i> .	Aqueous solution, 1:20.	"	" "	" "
<i>Zinc phenol-sulphonate</i> .	Aqueous solution, 1:20.	"	" "	" "
<i>Zinc sulphate</i> .	Aqueous solution, 1:20.	"	" "	" "
<i>Phosphoric acid</i> .	Aqueous solution, 1:5.	"	" "	" "
<i>Potassium permanganate</i> .	0.5 Gm. salt boiled with 20 Cc. water and 4 Cc. alcohol till decomposed. 5 Cc. of fil- is taken.	Yes.	" "	No precipitate nor cloudiness.
<i>Ammonia water</i> .	Supersaturated with nitric acid.		" "	Not affected.
<i>Ammonium benzoate</i> .	Aqueous solution, 1:10, treated with diluted nitric acid and filtrate taken.		" "	" "
<i>Ammonium carbonate</i> .	Aqueous solution, 1:20.	Added after AgNO ₃ T.S.	" "	Not more than opalescent after two minutes.
<i>Calcium phosphate</i> .	5 Cc. of nitric acid solution, 1:10.		0.5 Cc.	No turbidity.
<i>Zinc oxide</i> .	Nitric acid solution.		No mention.	Remains clear.
<i>Zinc valerate</i> .	0.5 Gm. salt dissolved in 0.5 Cc. nitric acid and 4.5 Cc. water and filtered through wet paper.		" "	Not more than faint cloudiness.
<i>Ferric ammonium sulphate</i> .	30 Cc. aqueous solution, 1:20.	3 Cc. added.	Few drops.	No turbidity.
<i>Antimony and potassium tartrate</i> .	Aqueous solution, 1:100, acidulated with acetic acid.	None.	" "	No effect.

CHEMICAL.	STRENGTH OR CHARACTER OF SOLUTION.	NITRIC ACID ADDED?	AMOUNT SILVER NITRATE T.S. USED.		RESULT.
<i>Red mercuric iodide.</i>	Shake 0.5 Gm. salt with 10 Cc. water and use filtrate.	None.	No mention.		Not more than opalescent.
<i>Yellow mercuric oxide.</i>	0.1 Gm. dissolved in 10 Cc. diluted nitric acid.		“	“	Not more than opalescent.
<i>Red mercuric oxide.</i>					
<i>Solution of formaldehyde.</i>	5 Cc. solution diluted with 15 Cc. water.	None.	“	“	No turbidity nor precipitate.
<i>Acetic acid.</i>	Diluted with ten volumes water.	“	“	“	No turbidity nor precipitate.
<i>Glycerin.</i>	Aqueous solution, 1: 10.	“	“	“	No color, cloudiness, nor precipitate.
<i>Honey.</i>	Aqueous solution, 1: 5.	“	Few drops.		Not more than faintly opalescent.
<i>Phenyl salicylate.</i>	Filtrate after treatment of aqueous solution (1: 50) with ferric chloride T.S. (See p. 955.)	“	“	“	No turbidity.

Several of the chloride tests are too complicated to permit tabulation, and these are given in full below. It will be noticed that most of these are those given for detecting chlorides in bromides and iodides, these two classes of salts precipitating with silver nitrate as quickly as do chlorides.

Diluted Hydrobromic Acid.—If 0.5 Cc. of Diluted Hydrobromic Acid be mixed with 10 Cc. of water, and 8 Cc. of silver nitrate T.S. with 6 Cc. of ammonium carbonate T.S. be added, and if the mixture, after digesting for ten minutes on a bath of boiling water, be cooled and filtered, the filtrate, on supersaturating with nitric acid, should not become more than slightly opalescent (limit of *hydrochloric acid* and *chlorides*).

Potassium Iodide.—If 0.2 Gm. of Potassium Iodide be dissolved in 2 Cc. of ammonia water (10 per cent.), and 13 Cc. of tenth-normal silver nitrate V.S. be added, then, after thoroughly agitating and filtering, the filtrate, upon acidifying with nitric acid, should not become more than slightly turbid nor should any darkening appear within ten minutes (limit of *chlorides* and *bromides* and absence of *thiosulphate*).

Sodium iodide is tested like potassium iodide, except 13 Cc. tenth-normal silver nitrate solution is used and nitric acid is added to supersaturation.

Ammonium Iodide.—If 0.25 Gm. of the salt, dried at 100° C. (212° F.), be dissolved in 5 Cc. of ammonia water (10 per cent.), the solution shaken with 16.9 Cc. of tenth-normal silver nitrate V.S., and the filtrate supersaturated with 5 Cc. of nitric acid, no cloudiness should make its appearance within ten minutes (absence of more than about 3 per cent. of *chlorides* or *bromides*).

Zinc Bromide.—If 1 Gm. of Zinc Bromide be dissolved in 50 Cc. of acetic acid, 2 Gm. of lead dioxide (free from chloride) be added, and the mixture evaporated in a small beaker to at least 10 Cc., then the residue, diluted with 10 Cc. of distilled water and filtered, should give not more than a slight turbidity on the addition of 2 Cc. of nitric acid and a few drops of silver nitrate T.S. (limit of *chloride*).

Zinc Iodide.—If 1 Gm. of Zinc Iodide be mixed with 5 Cc. of distilled water, and sufficient ammonia water be added to redissolve the precipitate formed, followed by a solution of 1.5 Gm. of silver nitrate in 10 Cc. of water, then, after shaking and filtering, the filtrate should not be rendered more than slightly turbid by the addition of an excess of nitric acid (limit of *chloride*).

Zinc Stearate.—If 0.5 Gm. of Zinc Stearate be heated with a mixture of 9.5 Cc. of distilled water and 0.5 Cc. of nitric acid, and filtered, the filtrate should not become

more than slightly turbid upon the addition of silver nitrate T.S. (absence of more than traces of *chlorides*).

Bismuth Subcarbonate.—If a solution of 0.3 Gm. of the salt in 10 Cc. of diluted nitric acid be treated with 0.1 Cc. of tenth-normal silver nitrate V.S., and the precipitate, if any, removed by filtration, the clear filtrate should remain unaffected by the further addition of the reagent (limit of *chlorides*).

Bismuth Subnitrate.—Take solution of 3 Gm. of salt in 3 Cc. warm nitric acid, precipitate by pouring into 100 Cc. water, evaporate filtrate to 30 Cc., filter, and use 5 Cc. of this filtrate as in case of bismuth subcarbonate.

Bismuth Citrate.—

Bismuth and Ammonium Citrate.—

Bismuth Subgallate.—

Bismuth Subsalcicylate.—

} Dissolve ash from 3 Gm. salt in warm nitric acid, and proceed as with bismuth subcarbonate.

Lactic Acid.—Ten Cc. of a solution of the Acid in distilled water (1 in 100) should not be rendered opalescent by 1 Cc. of silver nitrate T.S. (limit of *chloride*).

Salicylic Acid.—A solution of 0.5 Gm. of the Acid in 10 Cc. of alcohol, mixed with a few drops of nitric acid, should remain unaffected upon the addition of a few drops of silver nitrate T.S. (absence of *hydrochloric acid*).

Before proceeding farther it is well to mention that the tests for purity apply as much to a diluted compound as to the stronger; thus, while in the above table diluted nitric acid and diluted sulphuric acid are not given, the chloride test applies as much to them as to the strong acids.

Hypochlorites evolve odor of chlorine when treated with an acid. This test is applied by the pharmacopœia in identifying *chlorinated lime* and *solution of chlorinated soda* (p. 393).

Chlorates, on heating with hydrochloric acid, evolve chlorine. On heating strongly, chlorates evolve oxygen. These tests are used for identifying *potassium chlorate* and *sodium chlorate*.

Sought as impurity in *potassium nitrate*.—

No yellow color should appear when 0.1 Gm. of the dry salt is sprinkled upon 1 Cc. of pure concentrated sulphuric acid (absence of *chlorate* and *perchlorate*).

Perchlorates.—Sought as impurity in *potassium nitrate*. See test just given.

Free bromine liberates iodine from potassium iodide.—(See chlorine test for hydrochloric acid, diluted hydrobromic acid, and nitric acid on p. 946.)

Hydrobromic acid and bromides give, with silver nitrate T.S., a yellowish-white precipitate insoluble in diluted nitric acid, but soluble in ammonia water. This precipitate is silver bromide (AgBr).

The following official chemicals are tested by this process:

PRESENT AS INGREDIENTS:

Diluted Hydrobromic Acid.—See p. 413.

Potassium Bromide.—

Sodium Bromide.—

Lithium Bromide.—

Ammonium Bromide.—

Calcium Bromide.—

Zinc Bromide.—

} Same as test for diluted hydrobromic acid.

Hyoscine Hydrobromide.—Also the test; chlorine water and shaking with chloroform turns the chloroform yellow.

Hyoscyamine Hydrobromide.—Same as test for diluted hydrobromic acid.

Quinine Hydrobromide.—On precipitating a saturated aqueous solution of the salt with sodium hydroxide T.S., filtering, supersaturating the filtrate with acetic

acid, adding chloroform and a little chlorine water, and shaking, the chloroform will separate with a yellow color.

Bromides sought as impurity in—

Sodium Iodide.— } Same test given for chloride (p. 948).
Potassium Iodide.— }

Sodium Chloride.—If 2 Gm. of the finely powdered salt be digested for some hours with 25 Cc. of warm alcohol, and, after cooling, the undissolved salt be removed by filtration, the filtrate evaporated to dryness, and the residue dissolved in 5 Cc. of water, and if 1 Cc. of chloroform be added, and chlorine water which has been diluted with twice its volume of water, cautiously introduced, drop by drop, with constant agitation, the chloroform should not acquire a violet, yellow, or orange color (absence of *bromide* and *iodide*).

Bromic acid and bromates liberate bromine (which colors chloroform brown yellow) when treated with strong acids or other chemicals. The following official chemicals are tested this way:

Sought as impurity in—

Nitric Acid.—If the diluted Acid (1 in 3) be shaken with a few drops of chloroform, the latter should remain colorless (absence of *iodine* or *bromine*), even after the introduction of a small piece of metallic tin (absence of *iodic* or *bromic acid*).

Potassium Bromide.—If diluted sulphuric acid be dropped upon crushed crystals of the salt, and the mixture be shaken with 1 Cc. of chloroform, the latter should not assume a yellowish-brown color (absence of *bromate*).

Sodium Bromide.—If diluted sulphuric acid be dropped upon some of the powdered salt, no yellow color should appear at once (absence of *bromate*).

Ammonium Bromide.—If a few drops of diluted sulphuric acid be brought in contact with a little of the powdered salt on a porcelain plate, the salt should not at once assume a yellowish color (absence of *bromate*).

Calcium Bromide.—Same as sodium bromide just given.

Free iodine gives deep blue color to starch mucilage, which color is discharged on addition of sodium thiosulphate solution. It also transmits to chloroform a violet color. The following official chemicals are tested this way.

PRESENT AS INGREDIENT:

Iodine.—See p. 395.

Sought as impurity in—

Hydrochloric Acid.—If to 5 Cc. of Hydrochloric Acid, diluted with an equal volume of water, 1 Cc. of chloroform be added, and if chlorine water which has been diluted with an equal volume of water be then cautiously added, a drop at a time, with constant agitation, the chloroform should remain free from any yellow, orange, or violet color (absence of *bromine* or *iodine*).

Diluted Hydrobromic Acid.—Practically same test as the one for hydrochloric acid just given.

Nitric Acid.—See bromic acid test for nitric acid given above.

Sodium Iodide.—See iodate test for sodium iodide on p. 951.

Lithium Bromide.—Exactly same as iodide test for potassium bromide (p. 951), except full strength chlorine water is used.

Ammonium Iodide.—No violet color on shaking aqueous solution (1:100) with chloroform.

Thymol Iodide.—If 0.5 Gm. be shaken with 10 Cc. of water, and the mixture filtered, the filtrate should not be colored blue upon the addition of starch T.S. (absence of *free iodine*).

Hydriodic Acid and Iodides.—Chlorine water liberates free iodine, which gives violet color to chloroform. Silver nitrate gives yellow precipitate, insoluble in ammonia water as well as in hydrochloric acid. This precipitate is silver iodide (AgI).

The following official chemicals are tested in this way:

PRESENT AS INGREDIENTS:

Diluted Hydriodic Acid.—See p. 415.

Potassium Iodide.—If to 5 Cc. of the aqueous solution of the salt (1 in 20) 1 Cc. of chlorine water be added, iodine will be liberated, and impart to the solution a light reddish-brown color. On agitating the mixture with a few drops of chloroform, the latter will acquire a violet color.

Sodium Iodide.—

Ammonium Iodide.—

Strontium Iodide.—

Zinc Iodide.—Gives, with silver nitrate T.S., a pale yellow precipitate insoluble in ammonia water.

} Exactly as the potassium iodide test just given.

Sought as impurity in—

Potassium Bromide.—If to 10 Cc. of the aqueous solution of the salt (1 in 20) 1 Cc. of chloroform be added, and if chlorine water, which has been diluted with an equal volume of water, be introduced cautiously, drop by drop, with constant agitation, the liberated bromine will dissolve in the chloroform, imparting to it a yellow to orange color, free from any violet tint (absence of *iodides*).

Potassium Nitrate.—Like potassium bromide test just given, except, of course, no bromine is liberated.

Sodium Bromide.—Exactly like potassium bromide test just given.

Sodium Chloride.—See bromide test for sodium chloride on p. 950.

Sodium Nitrate.—Exactly like potassium nitrate test just given.

Ammonium Bromide.—

Calcium Bromide.—

Strontium Bromide.—

Zinc Bromide.—

Red Mercuric Iodide.—If about 0.5 Gm. of Mercuric Iodide be thoroughly agitated with 10 Cc. of distilled water, the filtered liquid should not become more than slightly colored by hydrogen sulphide T.S. nor give more than a slight opalescence with silver nitrate T.S. (limit of soluble *chlorides* or *iodides*).

Thymol Iodide.—If 0.5 Gm. be shaken with 10 Cc. of water, the mixture, on filtering, will yield a filtrate which should not become more than opalescent on the addition of nitric acid and silver nitrate T.S. (absence of *iodides*).

Iodic Acid and Iodates.—Iodine is liberated by action of acids and other chemicals, transmitting a yellowish-brown color to solution. The following official chemicals are tested this way:

Sought as impurity in—

Nitric Acid.—See bromic acid test for nitric acid on p. 950.

Potassium Iodide.—If to 0.5 Gm. of the salt, dissolved in 10 Cc. of distilled water, which has previously been boiled and cooled in a small flask, 2 drops of diluted sulphuric acid (free from sulphurous and nitrous acids) be added, no distinct yellow color should appear within half a minute (limit of *iodate*).

Sodium Iodide.—If 0.5 Gm. of the salt be dissolved in 10 Cc. of distilled water, which has been previously boiled and cooled in a small flask, the solution should not have a distinct yellow tint (absence of *free iodine*), nor should the solution acquire a yellow color within half a minute after the addition of 2 drops of diluted sulphuric acid (which should be free from sulphurous acid or nitrous acid) (limit of *iodate*).

Hydrofluoric acid and fluorides give off vapors which etch glass. (Fluorides must be warmed with sulphuric acid.)

Sought as impurity in—

Solution of Hydrogen Dioxide.—On evaporating to dryness, upon a water-bath, 50 Cc. of the Solution, previously rendered alkaline by sodium hydroxide T.S., transferring the dry residue to a watch-glass, moistening it with sulphuric acid, and setting the glass in a moderately warm place for a few hours, the surface of the glass, after being washed, should exhibit no sign of corrosion (absence of *hydrofluoric acid*).

Sulphides.—When treated with a diluted acid give off hydrogen sulphide gas, characterized by its bad smell and its ability to blacken paper moistened with lead acetate T.S. See sulphurated lime (p. 508).

Present as impurity in—

Sodium Thiosulphate.—Five Cc. of 5 per cent. aqueous solution give no black precipitate with one drop of silver nitrate T.S.

Sulphurous acid and sulphites when heated (*the sulphites when treated with a diluted acid*) give off sulphur dioxide gas, characterized by its suffocating odor and its ability to blacken paper moistened with mercurous nitrate, T.S. When treated with zinc and hydrochloric acid, they evolve hydrogen sulphide gas.

The following official chemicals are tested this way:

PRESENT AS INGREDIENTS:

Sulphurous Acid.—See p. 425.

Sodium Bisulphite.—On the addition of hydrochloric or sulphuric acid, an aqueous solution of the salt evolves sulphur dioxide, which is recognized by its odor and by its blackening a strip of paper dipped into mercurous nitrate T.S. and held over the escaping gas.

Sodium Sulphite.—Same as sodium bisulphite test just given.

Sought as impurity in—

Hydrochloric Acid.—No turbidity on adding a few drops of tenth-normal iodine V.S. to a mixture of the acid with barium chloride T.S.

Sulphuric Acid.—See test for nitrous acid in sulphuric acid (p. 955).

Sodium Salicylate.—If to an aqueous solution of the salt (1 in 20), 3 drops of iodine T.S. and a slight excess of hydrochloric acid be added, the filtrate from this mixture should not yield a precipitate upon the addition of barium chloride T.S. (absence of *sulphites*).

This test, as well as the hydrochloric acid test outlined above, is based on the property of iodine to convert sulphurous acid into sulphuric acid.

Sodium Thiosulphate.—See thiosulphate test under sodium thiosulphate.

Sulphuric Acid and Sulphates.—*Barium chloride T.S. gives a white precipitate, insoluble in hydrochloric acid.* This precipitate is barium sulphate (BaSO_4). The following official chemicals are tested this way:

PRESENT AS INGREDIENTS:

Sulphuric Acid.—See p. 422.

Potassium Sulphate.—An aqueous solution of the salt (1 in 20) yields with barium chloride T.S. a heavy white precipitate, insoluble in hydrochloric acid.

Sodium Sulphate.—Same as test for potassium sulphate just given.

Magnesium Sulphate.—Same as test for potassium sulphate just given, except strength of solution not mentioned.

Exsiccated Calcium Sulphate.—Saturated aqueous solution forms a white precipitate with barium chloride T.S.

Zinc Sulphate.—Same as test for potassium sulphate, given above, except no mention of insolubility in hydrochloric acid.

<i>Manganese Sulphate.</i> —	} Same as test for magnesium sulphate given on p. 952.
<i>Aluminum Sulphate.</i> —	
<i>Alum.</i> —	
<i>Ferrous Sulphate.</i> —	
<i>Ferric Ammonium Sulphate.</i> —	
<i>Solution of Ferric Sulphate.</i> —	} Same as test for potassium sulphate given on p. 952.
<i>Solution of Ferric Subsulphate.</i> —	
<i>Copper Sulphate.</i> —	
<i>Sparteine Sulphate.</i> —	
<i>Hyoscyamine Sulphate.</i> —	
<i>Quinine Bisulphate.</i> —	} Same as test for magnesium sulphate given on p. 952.
<i>Quinine Sulphate.</i> —	
<i>Cinchonidine Sulphate.</i> —	
<i>Strychnine Sulphate.</i> —	
<i>Morphine Sulphate.</i> —	
<i>Codeine Sulphate.</i> —	} Same as test for potassium sulphate given on p. 952, except strength of solution is 1: 100.
<i>Cinchonine Sulphate.</i> —	

Sought as impurity in—

CHEMICAL.	STRENGTH OR CHARACTER OF SOLUTION.	ACID ADDED.	AMOUNT BaCl ₂ T.S. USED.	RESULT.
<i>Hydrochloric acid.</i>	Aqueous solution, 1: 6.	None.	Few drops.	No precipitate nor turbidity in an hour.
<i>Phosphoric acid.</i>	Aqueous solution, 1: 6.	"	No mention.	No precipitate.
<i>Diluted hydrobromic acid.</i>	U. S. P. acid.	"	" "	No precipitate nor turbidity.
<i>Diluted hydriodic acid.</i>	U. S. P. acid.	"	" "	No precipitate nor turbidity.
<i>Boric acid.</i>	Aqueous solution, 1: 25.	"	" "	No precipitate.
<i>Nitric acid.</i>	Aqueous solution, 1: 10.	"	" "	" "
<i>Sulphurous acid.</i>	10 Cc. U.S. P. acid.	1 Cc. diluted hydrochloric acid.	1 Cc.	Not more than slight turbidity.
<i>Hypophosphorous acid.</i>	Acid neutralized with ammonia water.	None.	No mention.	Not more than slight turbidity.
<i>Potassium permanganate.</i>	Prepared as in test for chlorides, p. 947.	Acidulated with nitric acid.	" "	Not more than slight turbidity.
<i>Ammonia water.</i>	Slightly supersaturated with nitric acid.	Yes.	" "	Not affected.
<i>Ammonium benzoate.</i>	Prepared as in test for chlorides (p. 947).	"	" "	" "
<i>Ammonium carbonate.</i>	Aqueous solution, 1: 20, slightly supersaturated with hydrochloric acid.	"	" "	" "
<i>Ammonium chloride.</i>	Aqueous solution, 1: 20.	None.	" "	" "
<i>Zinc acetate.</i>	Aqueous solution, 1: 20.	Few drops diluted nitric acid.	" "	Should remain clear.
<i>Zinc bromide.</i>	Aqueous solution, 1: 20.	None.	No mention.	Not turbid.
<i>Zinc chloride.</i>	Aqueous solution, 1: 20.	1 Cc. diluted hydrochloric acid.	" "	Should remain clear.
<i>Zinc iodide.</i>	Aqueous solution, 1: 20.	None.	No mention.	Not turbid.

CHEMICAL.	STRENGTH OR CHARACTER OF SOLUTION.	ACID ADDED.	AMOUNT BaCl ₂ T.S. USED.	RESULT.
<i>Zinc oxide.</i>	1 Gm. dissolved in just enough diluted nitric acid.	Yes.	No mention.	Not more than slightly turbid.
<i>Zinc phenol-sulphonate.</i>	Aqueous solution, 1:20.	No.	" "	Not turbid.
<i>Zinc valerate.</i>	Prepared as in test for chlorides (p. 947).	Yes.	" "	Only faint cloudiness.
<i>Aluminum hydroxide.</i>	1 Gm. in 20 Cc. diluted hydrochloric acid.	"	" "	Only faint cloudiness.
<i>Chromium trioxide.</i>	1 Gm. in 100 Cc. water, previously acidulated with a few Cc. hydrochloric acid.	"	1 Cc.	Not turbid.
<i>Saccharated ferrous carbonate.</i>	1 Gm. dissolved in 5 Cc. hydrochloric acid and diluted with water to 50 Cc.	"	No mention.	Not more than faint cloudiness.
<i>Antimony and potassium tartrate.</i>	Aqueous solution, 1:100, acidulated with acetic acid.	"	Few drops.	Not affected.
<i>Bismuth subnitrate.</i>	Prepared as in test for chlorides (p. 949).	"	Barium nitrate T.S. used.	" "
<i>Bismuth subcarbonate.</i>	Prepared as for chloride test for bismuth subnitrate (p. 949), except 4 Cc. warm nitric acid used.	"	Barium nitrate T.S. used.	" "
<i>Bismuth citrate.</i>	} Prepared as in test for chlorides (p. 949).	"	Barium nitrate T.S. used.	" "
<i>Bismuth and ammonium citrate.</i>				
<i>Bismuth subgallate.</i>				
<i>Bismuth subsalicylate.</i>				
<i>Solution of formaldehyde.</i>	Prepared as in test for chlorides (p. 948).	No.	Barium chloride T.S. No mention of amount.	No turbidity nor precipitate.
<i>Acetic acid.</i>	Diluted with 10 volumes of water.	"	Barium chloride T.S. No mention of amount.	No turbidity nor precipitate.
<i>Lactic acid.</i>	10 Cc. aqueous solution (1:10).	"	1 Cc.	Unaffected.
<i>Glycerin.</i>	Aqueous solution, 1:10.	No.	No mention.	No color cloudiness nor precipitate.
<i>Tartaric acid.</i>	Aqueous solution, 1:10.	Few drops hydrochloric acid.	" "	Unaffected.
<i>Citric acid.</i>	10 Cc. aqueous solution, 1:100.	Few drops hydrochloric acid.	1 Cc.	No turbidity.
<i>Honey.</i>	Aqueous solution, 1:4.	No.	No mention.	No opalescence.

CHEMICAL.	STRENGTH OR CHARACTER OF SOLUTION.	ACID ADDED.	AMOUNT BaCl_2 T.S. USED.	RESULT.
<i>Lemon juice.</i>	The filtered juice.	None.	No mention.	No turbidity nor white precipitate.
<i>Phenyl salicylate.</i>	Prepared as in test for chlorides (p. 948).	Few drops hydrochloric acid.	1 Cc.	No turbidity.
<i>Quinine hydrochloride.</i> <i>Quinine hydrobromide.</i>	Aqueous solution.	Few drops hydrochloric acid.	1 Cc.	Not more than faintly turbid.

Thiosulphates, on treatment with a strong acid, liberate sulphur dioxide, while sulphur precipitates from the solution. Other tests, see below. The following official chemicals are tested this way:

PRESENT AS INGREDIENT:

Sodium Thiosulphate.—Addition of sulphuric or hydrochloric acid to the aqueous solution liberates from it sulphur dioxide (recognized by its odor and by its blackening a strip of paper moistened with mercurous nitrate T.S. held in the escaping gas); a white precipitate of sulphur is also formed (distinction from *sulphite* or *bisulphite*).

Sought as impurity in—

Potassium Iodide.—See test for chlorides in potassium iodide (p. 948).

Sodium Bisulphite.—If 1 Gm. of Sodium Bisulphite be dissolved in 10 Cc. of diluted nitric acid, and the solution heated sufficiently to expel the gases, the liquid should not become turbid (absence of *thiosulphate*).

Sodium Iodide.—See test for chlorides in sodium iodide (p. 948).

Sodium Sulphite.—Same as test for sodium bisulphite given above.

Ammonium Carbonate.—Aqueous solution (1:20), on addition of slight excess of silver nitrate T.S. and supersaturation with nitric acid, should not become brown.

Nitrous acid and nitrites liberate iodine from potassium iodide with evolution of N_2O_4 gas; decolorize potassium permanganate when in acid solution, and are reduced to ammonia when treated with a metal and an acid. The following official chemicals are tested this way:

PRESENT AS INGREDIENT:

Sodium Nitrite.—If the aqueous solution of the salt be mixed with some potassium iodide T.S., and a few drops of an acid added, iodine will be liberated, and nitrogen dioxide gas will escape with effervescence.

Sought as impurity in—

Sulphuric Acid.—One Cc. of Sulphuric Acid, diluted with 5 Cc. of water and cooled, should not at once discharge the color of 0.1 Cc. of tenth-normal potassium permanganate V.S. (limit of *sulphurous* or *nitrous acid*).

Potassium Chlorate.—If to 1 Gm. of Potassium Chlorate contained in a test-tube of about 40 Cc. capacity, 5 Cc. of water, 5 Cc. of potassium hydroxide T.S., and about 0.2 Gm. of aluminum wire be added, and if in the upper portion of the test-tube a pledget of purified cotton be inserted, and over the mouth there be placed a piece of moistened red litmus-paper, then if the tube be heated upon a water-bath for fifteen minutes, no blue coloration of the paper should be discernible (limit of *nitrates* and *nitrites*).

Potassium Iodide.—
Sodium Iodide.— } Exactly as in testing potassium chlorate just given.

Nitric Acid and Nitrates.—The free acid discharges the color of indigo solution. The acid or a nitrate, plus sulphuric acid, converts green ferrous sulphate to red-brown ferric sulphate. If only a small

quantity is used and the solutions "layered" (see below), a black-brown ring is shown at point of contact of the two liquids. Other tests given below. The following official chemicals are tested this way:

PRESENT AS INGREDIENTS:

Nitric Acid.—See p. 416.

Potassium Nitrate.—If 5 Cc. of an aqueous solution (1 in 20) be agitated with an equal volume of sulphuric acid, and the liquid be cooled and a crystal of ferrous sulphate be placed in the liquid, a dark brown color should appear around the crystal.

If a drop of diphenylamine T.S. be mixed with 5 Cc. of an aqueous solution, and sulphuric acid be slowly added so as to form a separate layer, a deep blue color will appear at the line of contact.

Compound Solution of Sodium Phosphate.—The ferrous sulphate test just given.

Sodium Nitrate.—The diphenylamine test just given under potassium nitrate.

Lead Nitrate.— } On heating, the salt gives off nitrous vapors.

Silver Nitrate.— }

Mitigated Silver Nitrate.—The diphenylamine test just given under potassium nitrate.

Pilocarpine Nitrate.—If to an aqueous solution of the salt, mixed with an equal volume of ferrous sulphate T.S., sulphuric acid be carefully added without shaking, a brown ring will appear at the juncture of the two layers.

Strychnine Nitrate.—A solution of the salt, when poured carefully into a test-tube upon a layer of sulphuric acid containing in solution a little diphenylamine, will develop a blue zone at the juncture of the two liquids.

Sought as impurity in—

Phosphoric Acid.—If a crystal of ferrous sulphate be dropped into a cooled mixture of 1 Cc., each, of Phosphoric Acid, sulphuric acid, and water, no brown or brownish-black color should appear around the crystal (absence of *nitric acid*).

Similarly tested with crystal of ferrous sulphate are:

	STRENGTH AND CHARACTER OF SOLUTION OF SALT.	AMOUNT CONCENTRATED SULPHURIC ACID.	TIME LIMIT FOR BROWN COLORATION.
<i>Ferric Chloride</i> .—	Aqueous solution, 1: 10.	Equal volume.	Within five minutes.
<i>Solution of Ferric Chloride</i> .—			
<i>Tincture of Ferric Chloride</i> .—			
<i>Solution of Ferric Subsulphate</i> .—			
<i>Solution of Ferric Tersulphate</i> .—	Dilution, 1: 10.	Equal volume.	Immediately.
<i>Bismuth Citrate</i> .—			
	Filtrate after precipitating the metal with hydrogen sulphide.	Equal volume.	Immediately.
<i>Bismuth and Ammonium Citrate</i> .—	Prepared like bismuth citrate.	Equal volume.	Immediately.

Camphoric Acid.—If 2 Cc. of a saturated aqueous solution of Camphoric Acid be mixed with 2 Cc. of sulphuric acid in a test-tube, and 1 Cc. of a solution made by dissolving 1 Gm. of ferrous sulphate in 2 Cc. of diluted sulphuric acid be poured carefully upon it, no dark-colored zone should develop at the line of contact (absence of *nitric acid*).

Sulphuric Acid.—If there be carefully poured upon it, in a test-tube, a layer of ferrous sulphate T.S., and the liquid cooled, the zone of contact should not assume a brown or reddish color (limit of *nitric* or *nitrous acid*).

Potassium Carbonate.—If 5 Cc. of the aqueous solution (1 in 20) be carefully mixed with an equal volume of concentrated sulphuric acid, and, after cooling, 3 Cc.

of ferrous sulphate T.S. be poured upon it so as to form a separate layer, no brown color should appear at line of contact (absence of *nitrate*).

Red Mercuric Oxide.—If 1 Gm. of Red Mercuric Oxide be mixed with 5 Cc. of water and 2 Cc. of sulphuric acid added, the mixture cooled, and 2 Cc. of ferrous sulphate T.S. be carefully poured upon it, no brown zone should be developed at the line of contact upon standing (absence of *nitrate*).

Bismuth Subcarbonate.—If 0.05 Gm. of Bismuth Subcarbonate be agitated with 5 Cc. of a mixture of equal parts of water and ferrous sulphate T.S., and then cautiously poured over 5 Cc. of sulphuric acid (free from nitrous compounds), so as to form a layer above, no brownish-red zone should form at the line of contact of the two liquids (limit of *subnitrate*).

Bismuth Subgallate.—If 0.5 Gm. of Bismuth Subgallate be well mixed with 5 Cc. of diluted sulphuric acid, 5 Cc. of ferrous sulphate T.S. added, and this mixture cautiously poured, without shaking, over 5 Cc. of sulphuric acid (free from nitrous compounds) contained in a test-tube, no brown ring should form after standing for ten minutes (limit of *nitrate*).

Potassium Chlorate.—

Potassium Iodide.—

Sodium Iodide.—

Calcium Bromide.—

See test for nitrites in potassium chlorate (p. 955), only using 5 Cc. of 1 per cent. solution.

Lead Iodide.—See test for nitrites in potassium chlorate (p. 955), only using 5 Cc. of 2 per cent. solution.

Sodium Borate.—If 1 Gm. of the salt be dissolved in 20 Cc. of diluted sulphuric acid by the aid of heat, and 3 drops of indigo T.S. be added, the blue color should not be discharged after heating for ten minutes on a water-bath (absence of *nitrate*).

Bismuth Subsalcicylate.—If 0.2 Gm. of Bismuth Subsalcicylate be triturated with 0.3 Gm. of sodium salicylate and 5 Cc. of distilled water, and carefully poured, without mixing, over 5 Cc. of sulphuric acid (free from nitrous compounds) contained in a test-tube, no pink to brownish-red zone should form immediately (limit of *nitrates*).

Potassium Permanganate.—Five Cc. of the filtrate left after boiling salt with water and alcohol (p. 956) is submitted to the diphenylamine test, given under Potassium Nitrate.

Boric acid gives the alcohol flame a green tinge.

Borates give similar flame when treated with a few drops of concentrated sulphuric acid and a little alcohol and then ignited. The following official chemicals are tested in this way:

PRESENT AS INGREDIENTS:

Boric Acid.—Its solution in alcohol or glycerin, when ignited, burns with a flame enveloped with a green-colored mantle.

An aqueous solution of Boric Acid (1 in 50) colors blue litmus-paper red, and yellow turmeric paper brownish-red after drying, even when the solution has been acidulated with hydrochloric acid; this brownish-red color is changed to bluish-black by ammonia water.

Sodium Borate.—If a drop of the solution of Sodium Borate in glycerin be held in a non-luminous flame, a transient bright green color will appear.

If a slight excess of diluted sulphuric acid be added to a hot, saturated, aqueous solution of the salt, shining, scaly crystals of boric acid will separate on cooling, which, when dissolved in alcohol and the liquid ignited, impart a green color to the flame.

Hypophosphites act as reducing agents; that is, when in acid solution or when heated with solutions of salts of silver, copper, and mercury, the metal is eventually precipitated. On heating, hypophosphites yield the spontaneous combustible gas, phosphine (PH_3). (See p. 448.) The following official chemicals are tested in this way:

Potassium Hypophosphite.—The diluted aqueous solution, slightly acidulated with diluted sulphuric acid, yields, with silver nitrate T.S., a white precipitate, which rapidly turns brown or black, owing to the separation of metallic silver.

On gently heating an aqueous solution of Potassium Hypophosphite with copper sulphate T.S., a reddish-brown precipitate is formed.

When the aqueous solution of the salt (1 in 20), acidulated with hydrochloric acid, is added, drop by drop, to an excess of mercuric chloride T.S., a white precipitate of mercurous chloride is at first formed. On further addition of an excess of the Potassium Hypophosphite solution, the precipitate becomes gray from reduction to metallic mercury.

Sodium Hypophosphite.— } Same test as that for potassium hypophosphite just
Calcium Hypophosphite.— } given.

Manganese Hypophosphite.—The mercuric chloride test given under Potassium Hypophosphite.

Ferric Hypophosphite.—The silver nitrate and mercuric chloride tests given under Potassium Hypophosphite.

Phosphorous Acid.—*Reduce solution of silver nitrate and mercuric chloride as do hypophosphites. It also gives a white precipitate with barium chloride T.S.*

Sought as impurity in—

Phosphoric Acid.—If 1 Cc. of Phosphoric Acid be diluted with 5 Cc. of water, and the liquid gently warmed, it should not be blackened upon the addition of a few drops of silver nitrate T.S., or rendered turbid by mercuric chloride T.S. (absence of phosphorous acid).

Hypophosphorous Acid.—This acid should not turn turbid when barium chloride T.S. is added.

Phosphoric acid and phosphates give white, crystalline precipitate with magnesium mixture,¹ a yellow precipitate, soluble in ammonia and nitric acid with silver nitrate T.S., a yellow precipitate on warming with ammonium molybdate T.S. The acid differs from metaphosphoric acid in coagulating albumin; from hypophosphorous acid in giving a yellow precipitate with silver nitrate. The following official chemicals are tested in this way:

PRESENT AS INGREDIENTS:

Phosphoric Acid.—See p. 426.

Sodium Phosphate.—An aqueous solution of the salt (1 in 20) yields a white, crystalline precipitate with magnesia mixture T.S.

With silver nitrate T.S. an aqueous solution yields a yellow precipitate, soluble in ammonia water and in nitric acid.

If 0.5 Cc. of the aqueous solution (1 in 20) be mixed with 1 Cc. of ammonium molybdate T.S., the mixture will at once assume a yellow color, and after being gently heated a few minutes will yield a yellow precipitate.

Compound Solution of Sodium Phosphate.—If to 1 Cc. of the Solution, diluted with an equal volume of water, a slight excess of ammonia water, followed by 1 Cc. of magnesia mixture T.S., be added, a white, crystalline precipitate will be obtained.

Precipitated Calcium Phosphate.—Nitric acid solution, on addition of ammonium molybdate T.S., gives yellow precipitate.

Soluble Ferric Phosphate.—Dissolve 0.1 Gm. of the salt in 2 Cc. of warm water; add 5 Cc. of potassium hydroxide T.S., boil and filter. Neutralize the filtrate with hydrochloric acid, add 2 Cc. of magnesia mixture T.S. and ammonia water in slight excess. A white, crystalline precipitate is produced, which, if well washed on a filter, is turned yellow by silver nitrate T.S.

Codeine Phosphate.—The silver nitrate test. See Sodium Phosphate, just above.

Sought as impurity in—

Hypophosphorous Acid.—Same as test for phosphorous acid in hypophosphorous acid (see above).

¹*Magnesia Mixture T.S.*—Dissolve 10 Gm. magnesium sulphate and 20 Gm. ammonium chloride in 80 Cc. distilled water; add 42 Cc. ammonia water, U. S. P.; set aside a few days, and then filter.

Potassium Bitartrate.—If 1 Gm. of Potassium Bitartrate be well triturated with about 1 Gm. of potassium carbonate and 0.5 Gm. of potassium nitrate, and the mixture heated gradually to dull redness in a porcelain crucible, and if, upon cooling, the resulting mass be treated with a slight excess of diluted hydrochloric acid and filtered, the filtrate, upon being made slightly alkaline with potassium hydroxide T.S., should not yield a gelatinous precipitate soluble in excess of the reagent (absence of *alum*). If a precipitate be produced which is insoluble, it should be collected and thoroughly washed with hot distilled water and dissolved in hot diluted nitric acid; the addition of an excess of ammonium molybdate T.S. to this solution should not produce a yellow precipitate (absence of *phosphates*).

Sodium Borate.—Aqueous solution (1 in 20) not rendered turbid by magnesia mixture.

Sodium Pyrophosphate.—Gives white—not yellow—precipitate with silver nitrate T.S.

Manganese Hypophosphite.—If 0.25 Gm. of the salt be boiled with 10 Cc. of potassium hydroxide T.S., a light salmon-colored precipitate will be produced which gradually acquires a brown color on exposure to the air, and if the filtered liquid, after being slightly acidulated with hydrochloric acid and then rendered alkaline with ammonia water, be divided into two portions, one should yield no precipitate upon the addition of magnesia mixture T.S. (absence of *phosphate*).

Ferric Hypophosphite.—If 0.5 Gm. of the salt be boiled with 10 Cc. of potassium hydroxide T.S., a reddish-brown precipitate will be produced; and if to the filtrate from the latter, slightly acidulated with hydrochloric acid, magnesia mixture T.S. be added, and subsequently an excess of ammonia water, no crystalline precipitate should be produced (absence of *phosphate*).

Phosphates in phosphoric acid detected by solubility. The acid dissolves in three volumes of alcohol and one volume of ether. Phosphates do not.

Acid calcium phosphate detected in precipitated calcium phosphate by the fact that the latter turns yellow with silver nitrate T.S., while the acid phosphate stays white.

Metaphosphoric Acid.—See p. 426.

Pyrophosphates give a white precipitate with silver nitrate T.S.

PRESENT AS INGREDIENTS:

Sodium Pyrophosphate.—White precipitate with silver nitrate, soluble in ammonia water and nitric acid.

Soluble Ferric Pyrophosphate.—If 0.1 Gm. of the salt be fused with 0.1 Gm. each of potassium nitrate and sodium carbonate, and the residue boiled with 10 Cc. of distilled water and filtered, the filtrate, after being rendered nearly, but not quite, neutral with highly diluted nitric acid, should yield a yellow precipitate upon the addition of silver nitrate T.S.

When so treated, ferric pyrophosphate is converted into sodium phosphate, hence the yellow precipitate.

Pyrophosphoric acid is sought as impurity in—

Phosphoric Acid—Solution 1:6 should have no precipitate, even after several hours, on addition of an equal volume of the official tincture of ferric chloride. Also see p. 426.

Carbonic acid and **carbonates** effervesce on addition of a diluted acid. They give white precipitates with lime water. The following official chemicals are tested in this way:

PRESENT AS INGREDIENTS:

Monohydrated Sodium Carbonate.—See p. 460.

Sodium Bicarbonate.—See p. 464.

Magnesium Carbonate.—See p. 499.

Prepared Chalk.—See p. 510.

Precipitated Calcium Carbonate.—See p. 510.

Precipitated Zinc Carbonate.—See p. 521.

Bismuth Subcarbonate.—See p. 596.

Potassium Carbonate.—Aqueous solution (1:10) effervesces with acids.

Sought as impurity in—

Potassium Hydroxide.—On adding an excess of diluted sulphuric acid to 10 Cc. of an aqueous solution of Potassium Hydroxide (1 in 10), no distinct effervescence should occur (limit of *carbonate*).

Similarly tested are the following:

CHEMICAL.	ACID USED.	AMOUNT OF ACID USED.	STRENGTH OF SALT SOLUTION.	AMOUNT OF SALT SOLUTION.	RESULTS.
<i>Potassium cyanide</i> .	Diluted hydrochloric.	No mention.	1: 20.	No mention.	No effervescence.
<i>Potassium ferrocyanide</i> .	Diluted sulphuric.	No mention.	Concentrated solution.	No mention.	No effervescence.
<i>Potassium hypophosphite</i> .	Any.	No mention.	1: 20.	No mention.	No effervescence.
<i>Sodium hydroxide</i> .	Diluted sulphuric.	Excess.	1: 10.	10 Cc.	No distinct effervescence.
<i>Sodium borate</i> .	Any.	No mention.	1: 20.	No mention.	No effervescence.
<i>Sodium phosphate</i> .	Hydrochloric or nitric.	No mention.	No mention.	No mention.	No effervescence.
<i>Sodium pyrophosphate</i> .	Nitric.	No mention.	Precipitate obtained by treating 5 per cent. solution with silver nitrate T.S.	No mention.	No effervescence.
<i>Exsiccated calcium sulphate</i> .	A diluted.	No mention.	Dry salt.	No mention.	No effervescence.
<i>Manganese hypophosphite</i> .	Acetic.	5 Cc.	Dry salt.	0.5 Gm.	No effervescence.
<i>Ferric hypophosphite</i> .	Acetic.	10 Cc.	Dry salt.	1.0 Gm.	No effervescence.

Sodium Citrate.—The aqueous solution (1 in 20) should not be colored red by a drop of phenolphthalein T.S., nor effervesce on the addition of an acid (absence of *carbonate*).

Sodium Hypophosphite.— } Same as test for sodium citrate just given.
Sodium Thiosulphate.— }

Potassium Bicarbonate.—If 1 Gm. of the salt be dissolved without agitation in 20 Cc. of water at a temperature not above 15° C. (59° F.), and 0.2 Cc. of normal hydrochloric acid V.S. and 2 drops of phenolphthalein T.S. be added, a red tint should not appear immediately (limit of *carbonate*).

Sodium Bicarbonate.—Same as test for potassium bicarbonate just given.

Ammonia Water.—No turbidity with four volumes of lime water.

Magnesium Oxide.—If a mixture of 0.1 Gm. of Magnesium Oxide with 5 Cc. of water be heated to boiling, and, after cooling, be poured into 5 Cc. of acetic acid, solution should take place without the evolution of more than a few isolated gas bubbles (limit of *carbonate*).

Lime.—If 1 Gm. of Calcium Oxide be slaked and then thoroughly mixed with 50 Cc. of water, and the greater portion of the milky liquid decanted, the addition of hydrochloric acid to this residue should not cause more than a slight effervescence (limit of *carbonate*).

Solution of Lime.—The alkaline reaction of the Solution should entirely disappear, after it has been saturated with carbon dioxide, and subsequently boiled (absence of *alkalis* and their *carbonates*).

Cerium Oxalate.—No effervescence should occur when the salt is dissolved in diluted hydrochloric acid.

Silicates.—*Soluble silicates* (those of the alkaline metals), when treated with sulphuric acid, give a gelatinous precipitate of silicic acid, $\text{Si}(\text{OH})_4$. Soluble silicates can be precipitated from aqueous solution by addition of alcohol.

Sought as impurity in—

Sodium Hydroxide.—If 0.7 Gm. of Sodium Hydroxide be dissolved in 1.5 Cc. of water, and the solution added to 10 Cc. of alcohol, not more than a slight, white precipitate should occur within ten minutes (limit of *silicate*, etc.).

Permanganates give rose-colored aqueous solution and the color is discharged on addition of an oxidizable substance.

PRESENT AS INGREDIENT:

Potassium Permanganate.—See p. 451.

ORGANIC ACIDS

Acetic acid and acetates, when warmed with alcohol and sulphuric acid, give off odor of acetic ether. Neutral acetates give deep red color with ferric chloride. The following official chemicals are thus tested:

PRESENT AS INGREDIENT:

Acetic Acid.—See p. 671.

Potassium Acetate.—If a few particles of the salt be added to a mixture of 1 Cc. of sulphuric acid and 1 Cc. of alcohol, acetic ether will be formed, recognizable by its odor.

The addition of a little ferric chloride T.S. to a solution of the salt produces a deep red color, and, upon boiling, a pale brown, flocculent precipitate of basic ferric acetate separates.

Sodium Acetate.—Both tests given under potassium acetate just above, except in the ferric chloride test the strength of the acetate solution (1 in 20) is given.

Zinc Acetate.—The addition of a few drops of ferric chloride T.S. to an aqueous solution of the salt (1 in 20) produces a red color.

Lead Acetate.—On heating the salt with sulphuric acid, vapors of acetic acid are evolved.

Sought as impurity in—

Ammonium Valerate.—If a neutral solution of the salt be completely precipitated by ferric chloride T.S., the filtrate should not possess a deep red color (absence of acetate).

Zinc Valerate.—If 0.5 Gm. of Zinc Valerate be triturated with 3 Cc. of water and 0.2 Cc. of ferric chloride T.S. added, the filtrate should not show a red color (absence of acetate).

Lead Iodide.—If 1 Gm. of Lead Iodide be boiled for a few minutes with 20 Cc. of water, the mixture then cooled and filtered, and the lead removed from the filtrate by hydrogen sulphide, a portion of the second filtrate, after boiling to drive off hydrogen sulphide and carefully neutralizing with ammonia water, should not be colored red by a drop of ferric chloride T.S. (absence of acetate).

Lemon Juice.—If an equal volume of sulphuric acid containing a few drops of alcohol be added to Lemon Juice, and the liquid heated, no odor of acetic ether should be developed (absence of acetic acid).

Oxalic acid and oxalates give white precipitate of calcium oxalate on treatment with the solution of a calcium salt. They also give a white precipitate with barium chloride, soluble in nitric acid. The following official chemicals are thus tested:

Hypophosphorous Acid.—Not more than a slight turbidity with barium chloride T.S.

Tartaric Acid.—An aqueous solution (1 in 10), in which the free Acid has been nearly but not entirely neutralized by ammonia water, should not be affected by calcium sulphate T.S. (absence of, and difference from, *oxalic acid*).

Glycerin.—Aqueous solution (1 in 10) should give neither cloudiness, color, nor precipitate with calcium chloride T.S.

Butyrates give a blue-green precipitate with copper acetate solution. The following official chemical is thus tested:

Sought as impurity in—

Zinc Valerate.—If a concentrated solution of copper acetate in water be added to a concentrated aqueous solution of Zinc Valerate, the mixture should remain perfectly clear (absence of *butyrate*).

Valerates.—*Aqueous solutions of valerates when treated with sulphuric acid liberate free valeric acid, which is identified by its characteristic odor.* The following official chemicals are thus tested:

Ammonium Valerate.—If a concentrated aqueous solution of Ammonium Valerate be slightly supersaturated with sulphuric acid, an oily layer of valeric acid will rise to the surface.

Zinc Valerate.—See p. 527.

Tartaric acid and tartrates give white, crystalline precipitate of cream of tartar with addition of the proper quantity of potassium salt. When gently warmed with an ammoniacal solution of silver nitrate, the latter is reduced, and a silver mirror forms. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Tartaric Acid.—See p. 685.

Potassium Bitartrate.—In the aqueous solution of the salt, rendered neutral by potassium hydroxide T.S., silver nitrate T.S. produces a white precipitate, which, on boiling, becomes black through the separation of metallic silver. If, before applying heat, just sufficient ammonia water be added to dissolve the white precipitate, and the solution boiled, a mirror will be deposited on the sides of the test-tube.

Potassium and Sodium Tartrate.—With silver nitrate T.S. it produces a white precipitate, which becomes black on boiling.

Iron and Ammonium Tartrate.—If an aqueous solution of the salt (1 in 10) be deprived of its iron by boiling with an excess of potassium hydroxide T.S.; the filtrate, when slightly acidulated with acetic acid, will gradually deposit a white, crystalline precipitate.

Iron and Potassium Tartrate.—Test same as that for iron and ammonium tartrate just given.

Antimony and Potassium Tartrate.—On strong heat chars and gives odor of burnt sugar.

Sought as impurity in—

Hypophosphorous Acid.—Same test as that given for oxalates in hypophosphorous acid.

Potassium Citrate.—A solution of 1 Gm. of Potassium Citrate in 1 Cc. of water should not deposit any precipitate on the addition of 1 Cc. of acetic acid (absence of tartrate).

Ferric Citrate.—Same test as that given above for iron and potassium tartrate, except that no precipitate should be produced even after standing twenty-four hours.

Iron and Ammonium Citrate.—

Iron and Quinine Citrate.—

Soluble Iron and Quinine Citrate.—

Iron and Strychnine Citrate.—

} See Ferric Citrate just above.

Lemon Juice.—Upon the addition of solution of potassium acetate (1 in 3) and alcohol in excess, no white crystalline precipitate should form after allowing the liquid to stand fifteen minutes (absence of tartaric acid).

Citric acid and citrates, when boiled with calcium chloride T.S., deposit white granular precipitate of calcium citrate. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Citric Acid.—See p. 688.

Potassium Citrate.—On mixing 10 Cc. of the aqueous solution of the salt (1 in 20) with 10 Cc. of calcium chloride T.S., the liquid remains clear until it is boiled, when a white, granular precipitate is produced.

Sodium Citrate.—Like test for potassium citrate just given, except 2 Cc. calcium chloride T.S. is used.

Compound Solution of Sodium Phosphate.—If 5 Cc. of the Solution be neutralized with ammonia water, an excess of calcium chloride T.S. added, and the mixture filtered, the filtrate, upon boiling, should deposit a white precipitate.

Ferric Citrate.—If an aqueous solution of the salt (1 in 10) be deprived of its iron by boiling with an excess of potassium hydroxide T.S., and the filtrate be slightly acidulated with acetic acid, a portion of the cooled liquid, when mixed with a little calcium chloride T.S., and again heated to boiling, will gradually afford a white crystalline precipitate.

Iron and Ammonium Citrate.—

Iron and Quinine Citrate.—

Soluble Iron and Quinine Citrate.—

Iron and Strychnine Citrate.—

} See Ferric Citrate test just above.

Bismuth Citrate.—Filtrate, after treatment of a solution of 1 Gm. salt in ammonia water with hydrogen sulphide gas, boiled with excess of lime water, yields a white precipitate.

Bismuth and Ammonium Citrate.—Same as bismuth citrate test just given, except that sodium hydroxide T.S. is used instead of ammonia water.

Cyanides give a white precipitate with silver nitrate T.S., soluble in excess of cyanide solution. When treated with ferrous and ferric salts, alkali, and then an excess of acid, a blue color (ferric ferrocyanide) is produced. The following official chemicals are tested for cyanides:

PRESENT AS INGREDIENT:

Potassium Cyanide.—A few drops of a solution of the salt (1 in 20) yields with silver nitrate T.S. a white precipitate, which is soluble in an excess of the solution of Potassium Cyanide, also in ammonia water, and in concentrated nitric acid.

Sought as impurity in—

Potassium Iodide.—If 5 Cc. of the aqueous solution be gently heated with 1 drop of ferrous sulphate T.S., 1 drop of ferric chloride T.S., and 0.5 Cc. of potassium hydroxide T.S., no blue color should appear after acidulating the mixture with hydrochloric acid (absence of cyanide).

Sodium Iodide.—Same as potassium iodide test just given.

Ferrocyanides give deep blue precipitate with ferric salts. The following official chemicals are thus tested:

PRESENT AS INGREDIENT:

Potassium Ferrocyanide.—The color of the precipitate produced by the addition of ferric chloride T.S. to the diluted aqueous solution of the salt is dark blue; that produced by copper sulphate T.S. is reddish-brown; lead acetate T.S. or silver nitrate T.S. causes a white precipitate.

Sought as impurity in—

Potassium Cyanide.—Aqueous solution (1:20) acidulated with hydrochloric acid should not give blue color on addition of a drop of ferric chloride T.S.

Ferricyanides give deep blue precipitate with ferrous salts. The special test below employed by the pharmacopœia.

Sought as impurity in—

Potassium Ferrocyanide.—The precipitate produced by silver nitrate T.S. in the aqueous solution, acidulated with nitric acid, should be of a white color, without a tinge of red (absence of ferricyanide).

Sulphocyanates give intense red color with ferric salts. The following official chemicals are thus tested:

Sought as impurity in—

Potassium Cyanide.—Aqueous solution (1 in 20), acidulated with hydrochloric acid, should not give red color with a drop of ferric chloride T.S.

Sodium Bicarbonate.— } Like potassium cyanide test just given.
Ammonium Chloride.— }

Benzoates give flesh-colored precipitates with ferric salts. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Benzoic Acid.—See p. 779.

Sodium Benzoate.—If a few drops of ferric chloride T.S. be added to an aqueous solution of the salt, a salmon-colored precipitate will be deposited.

Lithium Benzoate.—Like sodium benzoate test just given, except strength of benzoate solution (1 in 20) is mentioned.

Ammonium Benzoate.—Like sodium benzoate test just given, except that saturated solution of the benzoate is employed.

Salicylates give violet color, or precipitate with ferric salts. When warmed with concentrated sulphuric acid and methyl alcohol, it gives the odor of methyl salicylate (oil of wintergreen). The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Salicylic Acid.—See p. 782.

Sodium Salicylate.—Ferric chloride T.S., added to an excess of a concentrated solution of the salt, produces a violet precipitate; but when added to a dilute solution (1 in 100) it produces a deep violet-blue color.

If copper sulphate T.S. be added to the aqueous solution (1 in 20) a green color is produced.

On adding to about 0.2 Gm. of the salt, in a test-tube, about 1 Cc. of concentrated sulphuric acid, and then, cautiously, drop by drop, about 1 Cc. of methyl alcohol, and heating the mixture to boiling, methyl salicylate will be evolved, recognizable by its odor.

Lithium Salicylate.—If a few drops of ferric chloride T.S. be added to an excess of a concentrated aqueous solution of Lithium Salicylate (1 in 4), a deep red color will be produced, which, after the liquid is largely diluted and mixed with more ferric chloride T.S., will change to a deep bluish-violet tint.

Upon adding to 0.5 Gm. of the salt, in a test-tube, about 1 Cc. of concentrated sulphuric acid, and cautiously, drop by drop, about 1 Cc. of methyl alcohol, then, on heating the mixture to boiling, the odor of oil of gaultheria will be evolved.

Ammonium Salicylate.—Like tests for sodium salicylate just given, except that the iron precipitate is dark red.

Strontium Salicylate.—Same as tests for sodium salicylate just given.

Bismuth Subsalicylate.—When Bismuth Subsalicylate is agitated with a solution of 5 drops of ferric chloride T.S. in 10 Cc. of water, a deep violet-blue coloration is produced.

Quinine Salicylate.—Its aqueous solution, when treated with a drop of ferric chloride T.S., should give a violet color.

Sulphuric acid containing about one-fifth of its volume of solution of formaldehyde gives a pink color.

Free salicylic acid is sought as impurity in—

Bismuth Subsalicylate.—If 1 Gm. of the salt be agitated with 5 Cc. of chloroform and the liquid filtered through a double filter of fine texture into 5 Cc. of water containing 3 drops of ferric chloride T.S., no violet zone should form within five minutes at the line of contact of the two liquids (absence of *free salicylic acid*).

Phenolsulphonates give pale violet color with ferric salts. Heated strongly, they char and give odor of phenol. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Sodium Phenolsulphonate.—A diluted solution of the salt (1 in 100) is rendered pale violet by ferric chloride T.S., but remains clear; barium chloride T.S. leaves the solution clear, but if a portion of the salt be ignited, and the residue dissolved in water, the same reagent will produce in the solution a copious white precipitate.

Zinc Phenolsulphonate.—A dilute solution of the salt (1 in 100) is colored pale violet by ferric chloride T.S.

Gallic acid and gallates give, with ferric chloride T.S., a blue-black color. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Gallic Acid.—See p. 788.

Bismuth Subgallate.—Filtrate after treatment of 0.1 Gm. chemical with hydrogen sulphide test solution gives blue-black coloration on addition of 1 drop ferric chloride T.S.

Free gallic acid is sought as impurity in—

Bismuth Subgallate.—If 0.5 Gm. of the salt be well shaken with 5 Cc. of alcohol and filtered at once, the filtrate should not turn moistened blue litmus-paper red (absence of *free gallic acid*).

Tannic acid and tannates give, with ferric chloride T.S., a blue-black precipitate. The acid differs from gallic acid yielding precipitates with solutions of alkaloids, glucosides, gelatin, albumin, and starch. The following official chemicals are thus tested:

PRESENT AS INGREDIENT:

Tannic Acid.—See p. 790.

Sought as impurity in—

Gallic Acid.—See p. 789.

THE METALS

Potassium and its salts give a violet tint to a colorless flame. When the proper quantity of tartaric acid or sodium bitartrate is added, a white crystalline precipitate (cream of tartar) results. When treated with platinic chloride T.S., a yellow, crystalline precipitate of potassiopluginic chloride is formed. When treated with sodio-cobaltic nitrite solution, a yellow precipitate falls. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Potassium Acetate.—The addition of sodium bitartrate T.S. to the concentrated aqueous solution of the salt causes a white, crystalline precipitate.

Potassium Bicarbonate.—See p. 437.

Potassium Bitartrate.—With sodium cobaltic nitrite T.S. the aqueous solution of the salt yields a copious yellow precipitate.

Potassium Bromide.—The addition of Tartaric acid T.S., or of sodium bitartrate T.S., to a concentrated aqueous solution of the salt produces a white crystalline precipitate.

Potassium Carbonate.—Its aqueous solution (1 in 10) yields with excess of tartaric acid T.S. a white crystalline precipitate.

Potassium Chlorate.—With excess of tartaric acid T.S., the solution (1 in 20) slowly yields a scant, white, crystalline precipitate; with platinic chloride T.S., a yellow crystalline precipitate is produced

Potassium Citrate.—

Solution of Potassium Citrate.— } Like test for potassium acetate given above.

Potassium Cyanide.—With an equal volume of sodium bitartrate T.S. the solution yields a white, crystalline precipitate.

Potassium Dichromate.—Like test for potassium bitartrate given above.

Potassium and Sodium Tartrate.—See p. 447.

Potassium Ferrocyanide.—Like test for potassium acetate given above.

Potassium Hydroxide.—See p. 433.

Potassium Hypophosphite.—Like test for potassium acetate given above.

Potassium Iodide.—Like test for potassium acetate given above, except that strength of iodide solution (1 in 20) is mentioned.

Potassium Nitrate.—Like test for potassium iodide just given.

Potassium Sulphate.—Like test for potassium acetate given above.

Alum.—When a saturated solution of Alum is shaken with sodium bitartrate T.S., it produces, within half an hour, a white, crystalline precipitate.

Iron and Potassium Tartrate.—If an aqueous solution of the salt (1 in 10) be deprived of its iron by boiling with an excess of potassium hydroxide T.S., the filtrate, when slightly acidulated with acetic acid, will gradually deposit a white, crystalline precipitate.

Mitigated Silver Nitrate.—Like test for potassium acetate given above, except the silver is removed with hydrochloric acid and the concentrated filtrate is used.

Sought as impurity in—

Hypophosphorous Acid.—Neither platinic chloride T.S. nor sodium cobaltic nitrite T.S. should produce more than a slight yellow turbidity in the diluted Acid (limit of *potassium*).

Sodium Hydroxide.—No precipitate on adding excess of tartaric acid T.S.

Sodium Acetate.—A saturated aqueous solution should not be rendered turbid by the addition of sodium bitartrate T.S. (limit of *potassium*).

Sodium Chlorate.—Like test for sodium acetate just given.

Sodium Iodide.—A solution of 1 Gm. of the salt in 1 Cc. of water should yield no precipitate with 1 Cc. of sodium bitartrate T.S. (limit of *potassium*).

Lithium Bromide.—If 0.5 Cc. of sodium cobaltic nitrite T.S. be added to 5 Cc. of an aqueous solution of the salt (1 in 20), no precipitate or turbidity should occur within 10 minutes (limit of *potassium*).

Sodium and its salts give a brilliant yellow tint to a colorless flame.
The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Sodium Hydroxide.—See p. 455.

Sodium Acetate.—

Sodium Arsenate.—

Sodium Arsenate, Exsiccated.—

Sodium Benzoate.—

Sodium Bicarbonate.—

Sodium Bisulphite.—

Sodium Borate.—

Sodium Bromide.—

Sodium Carbonate, Monohydrated.—

Sodium Chlorate.—

Sodium Chloride.—

Sodium Citrate.—

Sodium Hypophosphite.—

Sodium Iodide.—

Sodium Nitrate.—

Sodium Nitrite.—

Sodium Phenolsulphonate.—

Sodium Phosphate.—

Sodium Pyrophosphate.—

Sodium Salicylate.—

Sodium Sulphate.—

Sodium Sulphite.—

Sodium Thiosulphate.—

} Like test for sodium hydroxide (the yellow flame).

Lithium and its salts give crimson color to a non-luminous flame. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

<i>Lithium Benzoate.</i> —See p. 479.	
<i>Lithium Bromide.</i> —	
<i>Lithium Carbonate.</i> —	
<i>Lithium Citrate.</i> —	
<i>Lithium Salicylate.</i> —	

} Tested like lithium benzoate (crimson flame).

Ammonium salts, when heated with other alkalis, give off ammonia gas. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

<i>Ammonium Benzoate.</i> —See p. 487.	
<i>Ammonium Bromide.</i> —When the aqueous solution is gently heated with potassium hydroxide T.S., ammonia is evolved.	
<i>Ammonium Chloride.</i> —	
<i>Ammonium Iodide.</i> —	
<i>Ammonium Salicylate.</i> —	
<i>Ammonium Valerate.</i> —	

} Like test for ammonium bromide just given.

Ferric Ammonium Sulphate.—Like test for ammonium bromide just given, except that the potassium hydroxide solution gives a brown precipitate.

<i>Iron and Ammonium Tartrate.</i> —	
<i>Iron and Ammonium Citrate.</i> —	
<i>Soluble Iron and Quinine Citrate.</i> —	

} Like test for ferric ammonium sulphate just given.

Bismuth and Ammonium Citrate.—Like test for ammonium bromide given above.

Ammoniated Mercury.—When heated with potassium hydroxide T.S., the salt turns yellow and evolves vapor of ammonia.

Sought as impurity in—

Potassium Bitartrate.—The odor of ammonia should not be evolved on heating the salt with a slight excess of potassium hydroxide T.S.

Potassium and Sodium Tartrate.—Like test for potassium bitartrate just given.

Calcium Bromide.—See test for nitrates in Calcium Bromide on p. 957.

Alum.—Like test for potassium bitartrate given above, except that a white precipitate is formed.

Aluminum Sulphate.—If 1 Gm. of Aluminum Sulphate be gently heated with 5 Cc. of potassium hydroxide T.S., the liquid should not evolve the odor of ammonia.

Bismuth Subnitrate.—Like test for aluminum sulphate just given, except that 0.1 Gm. of salt is used.

Bismuth Subcarbonate.—Like test for aluminum sulphate just given, except that 10 Cc. of potassium hydroxide is used.

Magnesium salts give, with sodium phosphate, in the presence of ammonium chloride and ammonia water, a white, crystalline precipitate. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Magnesium Oxide.—See p. 497.

Magnesium Carbonate.—A filtered solution of the salt in diluted sulphuric acid, when mixed with ammonium chloride T.S. and an excess of ammonia water, yield, with sodium phosphate T.S., a white, crystalline precipitate.

Magnesium Sulphate.—Like test for magnesium carbonate just given.

Sought as impurity in—

Boric Acid.—Should not be precipitated by sodium phosphate T.S. and ammonia water.

Calcium compounds give, with ammonium oxalate, a white precipitate, insoluble in acetic acid, but soluble in hydrochloric acid. The following officials are thus tested:

PRESENT AS INGREDIENTS:

Chlorinated Lime.—From this solution (in diluted acetic acid) ammonium oxalate T.S. throws down a white precipitate, insoluble in acetic acid, but soluble in hydrochloric acid.

Calcium Bromide.—The aqueous solution of the salt (1 in 20) yields, with ammonium oxalate T.S., a white precipitate, insoluble in acetic acid, but soluble in hydrochloric acid.

Calcium Carbonate, Precipitated.—Like test for calcium bromide just given, except a solution is made with 5 Gm. salt, 100 Cc. water, and just enough hydrochloric acid.

Calcium Chloride.—Aqueous solution (1 in 20) tested like calcium bromide.

Calcium Hypophosphite.—Like test for calcium chloride just given.

Calcium Sulphate, Exsiccated.—Saturated aqueous solution, tested like calcium bromide.

Sought as impurity in—

Boric Acid.—Not precipitated by ammonium oxalate T.S.

Sodium Thiosulphate.—The aqueous solution (1 in 20) should not be rendered turbid by the addition of ammonium oxalate T.S. (absence of *calcium*).

Ammonium Chloride.—Not affected by ammonium oxalate T.S.

Magnesium Oxide.—Ten Cc. of a solution of Magnesium Oxide (1 in 50), prepared by the addition of sufficient acetic acid for solution, should not be rendered more than slightly opalescent within five minutes by ammonium oxalate T.S. (limit of *calcium*).

Magnesium Carbonate.—Ten Cc. of a solution of the salt (1 in 50), prepared by the addition of sufficient acetic acid for solution, should not be rendered more than slightly opalescent within five minutes by ammonium oxalate T.S. (limit of *calcium*).

Manganese Hypophosphite.—Filtrate obtained after boiling 0.25 Gm. salt with 10 Cc. potassium hydroxide T.S., acidulated with hydrochloric acid, and then made alkaline with ammonia water, is not affected by ammonium oxalate T.S.

Ferric Hypophosphite.—Solution of 1 Gm. salt in 10 Cc. acetic acid gives no turbidity with ammonium oxalate T.S.

Antimony and Potassium Tartrate.—One per cent. aqueous solution, acidulated with acetic acid, not affected by ammonium oxalate T.S.

Solution of Formaldehyde.—Twenty Cc. of aqueous dilution (1 in 4) should give neither turbidity nor precipitate on adding ammonium oxalate T.S.

Glycerin.—Aqueous solution (1 in 10) should give neither color, cloudiness, nor precipitate on adding ammonium oxalate T.S.

Tartaric Acid.—After supersaturating 10 Cc. aqueous solution (1 in 10) with ammonia water, not more than faint turbidity should be produced on adding ammonium oxalate T.S.

Citric Acid.—On mixing 5 Cc. of an aqueous solution of the Acid (1 in 10) with a quantity of ammonia water insufficient to neutralize it completely, and adding to this liquid 1 Cc. of ammonium oxalate T.S., it should remain clear (absence of *calcium*).

Strontium compounds give a crimson flame. Diluted sulphuric acid and solutions of sulphates give a white precipitate of strontium sulphate, which is soluble in strong acids. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Strontium Bromide.—See p. 515.

Strontium Iodide.—With calcium sulphate T.S. it slowly forms a white precipitate of strontium sulphate, insoluble in diluted acids; the same reaction occurs more quickly with diluted sulphuric acid and the readily soluble sulphates.

With potassium chromate T.S. it forms a yellow precipitate of strontium chromate, soluble in acetic acid.

With ammonium carbonate T.S., or sodium carbonate T.S., it forms a white precipitate of strontium carbonate, soluble, with effervescence, in acetic acid.

Strontium Salicylate.—Like test for strontium iodide just given.

Barium salts give a green flame. With potassium dichromate T.S. they give a yellow precipitate of barium chromate. With sulphuric acid they give a white precipitate of barium sulphate, insoluble in boiling nitric acid. The following official chemicals are thus tested:

Sought as impurity in—

Solution of Hydrogen Dioxide.—The addition of a few drops of diluted sulphuric acid to 10 Cc. of the Solution should produce no turbidity or precipitate (absence of *barium*).

Diluted Hydrobromic Acid.—Ten Cc. of the Acid should not be rendered turbid by the addition of 1 Cc. of potassium sulphate T.S. (absence of *barium*).

Diluted Hydriodic Acid.—The Acid should not be rendered turbid by the addition of potassium sulphate T.S. (absence of *barium*).

Hypophosphorous Acid.—Like test for diluted hydriodic acid, only first neutralize with ammonia water.

Potassium Bromide.—Like test for hydrobromic acid, except use of solution (1 in 20) acidulated with hydrochloric acid.

Potassium Iodide.—

Sodium Bromide.—

Sodium Iodide.—

} Like test for potassium bromide just given.

Ammonium Bromide.—Like test for potassium bromide just given, except acidulated with acetic acid.

Ammonium Chloride.—Not affected by diluted sulphuric acid.

Ammonium Iodide.—Like test for potassium bromide just given.

Calcium Bromide.—If 1 Gm. of Calcium Bromide and 1 Gm. of sodium acetate be dissolved in 5 Cc. of distilled water, and the solution made slightly acid through the addition of from 3 to 5 drops (or a sufficient quantity) of diluted acetic acid, after boiling and thoroughly cooling, the solution should not become cloudy within five minutes upon the addition, with agitation, of 5 drops of potassium dichromate T.S. (absence of *barium*).

Precipitated Calcium Phosphate.—Five Cc. of a solution made by mixing 2 Gm. salt with 20 Cc. water, nitric acid just enough to dissolve, then water to make 40 Cc., strongly acidulated with nitric acid, should give no turbidity on adding 1 Cc. potassium sulphate T.S.

Strontium Bromide.—

Strontium Iodide.—

} Same as test for calcium bromide given above, except

} does not cloud within three minutes.

Strontium Salicylate.—If 2 Gm. of Strontium Salicylate be agitated with 10 Cc. of diluted acetic acid, heated, cooled, and filtered, the filtrate, upon the addition of 5 drops of potassium dichromate T.S., should not become cloudy within three minutes (limit of *barium*).

Zinc salts give, with hydrogen sulphide T.S. or with ammonium sulphide T.S., a white precipitate of zinc sulphide; with potassium ferrocyanide, T.S., a white, gelatinous precipitate of zinc ferrocyanide. Heated with blow-pipe with cobaltous nitrate T.S. it turns a vivid green. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Zinc.—See p. 518.

Zinc Acetate.—The aqueous solution of the salt (1 in 20) yields, with potassium ferrocyanide T.S., a white, gelatinous precipitate, and with ammonium sulphide T.S., a pure white precipitate.

Zinc Bromide.—Like test for zinc acetate just given, except hydrogen sulphide T.S. also mentioned.

Precipitated Zinc Carbonate.—Like test for zinc acetate given above; using a solution of 1.25 Gm. of salt in 10 Cc. diluted sulphuric acid and 10 Cc. water. Also the cobaltous nitrate blow-pipe test.

Zinc Chloride.— } Like test for zinc acetate given above.
Zinc Iodide.— }
Zinc Oxide.—Like test for zinc acetate given above, using a solution of 1 Gm. oxide in 10 Cc. diluted hydrochloric acid and 10 Cc. water made by digestion.
Zinc Phenolsulphonate.—Like test for zinc acetate given above.
Zinc Sulphate.—Like test for zinc acetate given above; also cobaltous nitrate blow-pipe test.

Sought as impurity in—

Manganese Sulphate.—A solution of 1 Gm. each of the salt and of sodium acetate in 10 Cc. of water, to which a few drops of acetic acid have been added, should not be affected by hydrogen sulphide T.S. (absence of *zinc*).

Cerium Oxalate.—On boiling the salt with potassium hydroxide T.S. and filtering, no precipitate should be produced in the filtrate by the addition of ammonium sulphide T.S. (absence of *zinc*).

Manganese preparations, fused with alkaline hydroxides or carbonates and an oxidizing agent (potassium nitrate or chlorate), give a green mass (alkaline manganate), and this, when boiled with water, gives a purple solution (of alkaline permanganate). Solutions of its salts give, with ammonium sulphide T.S., a flesh-colored precipitate of manganese sulphide. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Precipitated Manganese Dioxide.—See p. 529.

Manganese Hypophosphite.—See p. 530.

Manganese Sulphate.—Yields, with ammonium sulphide T.S., a salmon-colored precipitate, soluble in dilute acids; with potassium ferrocyanide T.S., a reddish-white precipitate, and with potassium ferricyanide T.S., a brown precipitate.

Aluminum salts give, with ammonia water, a white, gelatinous precipitate of aluminum hydroxide, insoluble in excess of ammonia water; with potassa or soda they give the same precipitate, but it is soluble in excess of potassa or soda. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Alum.—Tested just as given above.

Aluminum Sulphate.—The aqueous solution of the salt yields, with potassium hydroxide T.S., a white, gelatinous precipitate which is soluble in an excess of the alkali, but which is again separated on the addition of a sufficient amount of ammonium chloride T.S.

Sought as impurity in—

Ferric Ammonium Sulphate.—If the iron be wholly precipitated from a solution of the salt by treating it with an excess of potassium hydroxide T.S., the resulting filtrate, when neutralized with hydrochloric acid and then mixed with ammonia water, should not yield a white, gelatinous precipitate (absence of *aluminum*).

Cerium Oxalate.—A potassium hydroxide filtrate (see test given under Zinc, p. 518) should give no precipitate on adding ammonium chloride T.S.

Potassium Bitartrate.—See test given under Phosphates (p. 959).

Copper Sulphate.—See p. 983.

Chromic acid salts, when warmed with sulphuric acid and alcohol, change to a green liquid which has the odor of aldehyde. The following official chemical is thus tested:

Potassium Dichromate.—On mixing 4 Cc. of an aqueous solution (1 in 20) with 0.5 Cc. of alcohol and then adding 1 Cc. of sulphuric acid, the liquid should assume a green color and emit the odor of aldehyde.

Ferrous salts give, with *potassium ferricyanide*, a blue color or precipitate. The following official salts are thus tested:

PRESENT AS INGREDIENTS:

Ferrous Sulphate.—The aqueous solution of the salt shows an acid reaction with blue litmus-paper, and even when highly diluted gives, with potassium ferricyanide T.S., a blue color or precipitate.

Saccharated Ferrous Carbonate.—If 1 Gm. of Saccharated Ferrous Carbonate be dissolved in 5 Cc. of hydrochloric acid, and the solution diluted with water until it measures 50 Cc., portions of this solution will yield a blue precipitate with both potassium ferrocyanide T.S. and potassium ferricyanide T.S.

This shows it contains both ferrous and ferric salt.

Sought as impurity in—

Ferric Chloride.—If to a solution of the salt (1 in 50) a few drops of freshly prepared potassium ferricyanide T.S. be added, a pure brown color should be produced, which should not turn at once to a green or greenish-blue (absence of *ferrous salt*).

Solution of Ferric Chloride.—Like test for ferric chloride just given, except a dilution 1 in 20 is used.

Solution of Ferric Sulphate.— } Like test for ferric chloride given above, except a dilution 1 in 10 is used.
Solution of Ferric Subsulphate.— }

Ferric salts (or iron) give a red-brown precipitate of ferric hydroxide with ammonia or soda or potassa. With *potassium ferrocyanide* they give a deep blue color or precipitate, and with *potassium sulphocyanate*, a blood-red color. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Ferric Chloride.—The dilute aqueous solution of the salt shows an acid reaction with blue litmus-paper, yields a brownish-red precipitate with ammonia water, and a blue precipitate with potassium ferrocyanide T.S.

Solution of Ferric Chloride.— } Like test for ferric chloride just given.
Tincture of Ferric Chloride.— }

Ferric Hypophosphite.—0.5 Gm. boiled with 10 Cc. potassium hydroxide T.S. gives reddish-brown precipitate.

Ferric Ammonium Sulphate.—The aqueous solution of the salt has a slightly acid reaction upon blue litmus-paper, and yields, with potassium ferrocyanide T.S., a blue precipitate.

With potassium hydroxide T.S. Ferric Ammonium Sulphate yields a brownish-red precipitate, and if the mixture be heated, ammonia gas is evolved.

Solution of Ferric Sulphate.— } Like test for ferric chloride just given.
Solution of Ferric Subsulphate.— }

Iron and Ammonium Tartrate.—With potassium ferrocyanide T.S. the solution does not yield a blue color or precipitate, unless it be acidulated with hydrochloric acid.

If Iron and Ammonium Tartrate be heated with potassium hydroxide T.S., it yields a brownish-red precipitate, and ammonia is evolved.

Iron and Potassium Tartrate.—Like test for iron and ammonium tartrate just given, except that no ammonia is evolved.

Ferric Citrate.—If Ferric Citrate be heated with potassium hydroxide T.S., it yields a brownish-red precipitate, without evolving ammonia.

Iron and Ammonium Citrate.—Like test for ferric citrate just given.

Iron and Quinine Citrate.—Filtrate after precipitating the quinine with ammonia does not turn blue with potassium ferrocyanide unless acidulated with hydrochloric acid. Also tested like ferric citrate, as just given.

Soluble Iron and Quinine Citrate.—Like tests for iron and quinine citrate, except that ammonia is evolved on heating with potassium hydroxide T.S.

Iron and Strychnine Citrate.—Like tests for soluble iron and quinine citrate just given.

Soluble Ferric Phosphate.— } Like tests for ferric citrate given above.
Soluble Ferric Pyrophosphate.— }

Sought as impurity in—

Boric Acid.—In a solution of 1 Gm. of Boric Acid in a mixture of 1 Cc. of hydrochloric acid and 49 Cc. of water, 0.5 Cc. of potassium ferrocyanide T.S. should not at once produce a blue color (limit of iron).

Ammonium Bromide.—An aqueous solution of the salt (1 in 150) should not at once assume a blue color with potassium ferrocyanide T.S. (limit of iron).

Ammonium Chloride.—Twenty Cc. of the aqueous solution of the salt (1 in 150) should not at once assume a blue color on the addition of 5 drops of potassium ferrocyanide T.S. (limit of iron).

Ammonium Iodide.—Like test for ammonium chloride given above.

Magnesium Oxide.—If 1 Gm. of Magnesium Oxide be dissolved in 50 Cc. of diluted hydrochloric acid, the solution should be colorless and not give an immediate blue coloration upon the addition of potassium ferrocyanide T.S. (limit of iron).

Magnesium Carbonate.—Like test for magnesium oxide just given, except a solution of 1 Gm. salt in 20 Cc. diluted hydrochloric acid is used.

Purified Talc.—Filtrate from 25 Cc. water, boiled with 5 Gm. talc, slightly acidulated with hydrochloric acid, should give no blue color with potassium ferrocyanide T.S.

Manganese Sulphate.—The aqueous solution of the salt (1 in 20), to which a few drops of hydrochloric acid and a few drops of chlorine water have been added, should, after having been boiled, not be colored red by potassium sulphocyanate T.S. (absence of iron).

Alum.—Like test for ammonium chloride given above.

Aluminium Hydroxide.—Like test for magnesium carbonate given above, except only one drop of ferrocyanide solution is to be used.

Aluminium Sulphate.—Like test for ammonium chloride given above.

Antimony and Potassium Tartrate.—One per cent. solution, acidulated with acetic acid, not affected by potassium ferrocyanide T.S.

Solution of Formaldehyde.—Twenty Cc. of aqueous dilution (1 in 4) should give neither turbidity nor precipitate on adding potassium ferrocyanide T.S. (This test for lead and copper as well as iron.)

Citric Acid.—See Heavy Metal Test (p. 982).

Sodium Arsenate.—See p. 983.

Copper Sulphate.—See p. 983.

Lead Acetate.—See p. 983.

Lead Nitrate.—See p. 983.

Arsenic and its compounds give, with hydrogen sulphide, a bright yellow precipitate of arsenic sulphide, which is soluble in ammonium carbonate T.S. and in ammonium sulphide T.S. With ammoniacal solution of copper sulphate¹ they give a green precipitate, while acid solutions dropped on a sheet of metallic copper coats the metal with a gray film of metallic arsenic. See also Marsh's Test (p. 573), Bettendorf's Test (p. 574 and below), and Gutzeit's Test (p. 574 and below). The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Arsenic Trioxide.—See p. 575.

Sodium Arsenate.—The aqueous solution of the salt (1 in 20) yields a white precipitate with barium chloride T.S. or with calcium chloride T.S., and a dark red precipitate with silver nitrate T.S., all of which precipitates are soluble in nitric acid.

If 0.5 Cc. of an aqueous solution (1 in 20) be mixed with 2 Cc. of hydrochloric acid, and a drop of this mixture be placed upon a bright piece of copper-foil, then upon applying a gentle heat a dark steel-gray film will be deposited upon the copper.

If to 2 Cc. of an aqueous solution (1 in 20), 5 Cc. of tenth-normal silver nitrate V.S. be added, and the precipitate redissolved by a slight excess of ammonia water, no black precipitate of reduced silver should appear on boiling (absence of arsenite).

¹ **Cupric Ammonium Sulphate Test Solution.**—A solution of cupri-tetrammonium sulphate, $\text{CuSO}_4 + 4\text{NH}_3 + \text{H}_2\text{O}$. To copper sulphate T.S. add ammonia water, until the precipitate first formed is nearly, but not completely, redissolved; then filter. This solution should be freshly made when required.

Sought as impurity in—

All official chemicals in which arsenic is to be sought as impurity are tested by one of two processes: the *modified Gutzeit test* or the *Bettendorf test*. The two tests as prescribed by the pharmacopœia are here reproduced, leaving the varieties of manipulation to be explained in tabulated form. In passing, it might be said that the extremely careful details of these tests are given by the pharmacopœia because the chemicals employed may possibly contain arsenic, hence must be most carefully tested.

Arsenic Test, Modified Gutzeit's.—The efficiency of this test depends upon a strict adherence to the conditions described below.

REAGENTS

Zinc.—This should be in a granulated or fine, mossy condition and free from arsenic, sulphur, and phosphorus, and should not contain more than 0.05 per cent. of iron. It should otherwise respond to all the tests given under Zinc (*Zincum*, U. S. P.).

Hydrochloric Acid (8 per cent.).—Mix 22.5 Cc. of hydrochloric acid (*Acidum Hydrochloricum*, U. S. P.) with water sufficient to measure 100 Cc.

Mercuric Chloride Test-paper.—By means of a glass rod, transfer a drop of a saturated alcoholic solution of mercuric chloride to the center of a piece of white filter-paper (preferably the kind used for quantitative analysis) of about 4 Cm. diameter; after drying, this paper is to be twice successively moistened with the reagent and dried. It is not necessary to moisten the paper with alcohol or the reagent while using it.

Lead Acetate Test-gauze.—A piece of cheese-cloth, about 1 decimeter square, when required for use, is thoroughly impregnated with lead acetate T.S., and the excess of fluid removed by pressure.

TEST-FLASK

Select a flask of the capacity of about 60 to 75 Cc., with a neck of about 5 to 6 Cm. in length and about 1 Cm. in diameter.

PREPARATION OF THE CHEMICAL TO BE TESTED

To 5 Cc. of the aqueous solution of the chemical (1 in 10) or to a solution in 5 Cc. of water of the residue remaining after undergoing special treatment, 1 Cc. of a mixture of equal volumes of sulphuric acid and water is added, followed by 10 Cc. of a freshly prepared saturated solution of sulphurous acid. This liquid, contained in a small beaker, is heated upon a bath of boiling water until it is free from excess of sulphurous acid and has been reduced to 5 Cc. in volume.

THE TEST

Before applying this test for the presence of arsenic in chemicals, in order to establish the freedom of the reagents from arsenic, sulphur, and phosphorus, or any interfering contaminations, a preliminary blank test should be made as follows: Into the flask are introduced 2 Gm. of zinc, 20 Cc. of the hydrochloric acid (see above), and 5 Cc. of water, and into the lower end of the neck of the flask is inserted a small wad of clean dry gauze, and then the lead acetate test-gauze, pressed with sufficient firmness to retain its place. About 1 Cm. space should be allowed above the gauze; the lip of the flask, after careful cleansing, is securely covered by folding over it the mercuric chloride test-paper. The reaction is allowed to proceed until the greater portion of the zinc has dissolved, which may require from one-half to two hours, when, if no trace of a yellow to orange-colored deposit is distinguishable upon the inner surface of the test-paper cap, the reagents are proved to be sufficiently pure, and a direct test may be applied at once. If a black stain is produced, sulphur compounds are present in the zinc or reagents, and this indicates unfitness for use. While the blank test is being carried out, another flask should be charged in a like manner with 2 Gm. of zinc and 20 Cc. of the hydrochloric acid (see above), followed by the 5 Cc. of the solution of the chemical (1 in 10) to be tested, which has undergone reduction as directed (under *Preparation of the Chemical to be tested*); the wad of clean dry gauze followed by the lead acetate test-gauze is then introduced, and after cleansing the lip of the flask the mercuric chloride cap is folded over the top. After the evolution of hydrogen has continued for at least one-half hour, and most of the zinc

has dissolved, the inner side of the mercuric chloride test-cap is examined to detect the presence of a yellow stain.

The presence of arsenic much in excess of the permissible limit (1 in 100,000) is manifested by the formation of a distinct yellow to orange spot, according to the quantity present. Antimony produces a dark gray to brownish-black coloration. The production of a black stain indicates the presence of sulphur compounds (as sulphurous acid or sulphides), also possibly antimony. If the former be present, a simultaneous blackening of the lead acetate gauze will be observed. If such be the case, the operation, as directed under *Preparation of the Chemical to be tested*, must be repeated upon a fresh portion of the sample, using greater precautions for the complete removal of the sulphurous acid.

In testing such phosphorus compounds as hypophosphorous acid and the hypophosphites, special care should be observed to completely oxidize the sample as directed, otherwise a yellow stain, similar to that caused by arsenic, may be produced through the evolution of hydrogen phosphide.

Compounds containing antimony are tested for arsenic by Bettendorf's Test.

The following chemicals, after preparation outlined below, are submitted to Gutzeit's test:

CHEMICAL.	PREPARATION FOR TESTING.	
<i>Solution of hydrogen dioxide.</i>	1 Cc. of solution mixed with 1 Cc. ammonia water and evaporated, dry residue,	Should not respond to Gutzeit's test.
<i>Washed sulphur.</i>	0.5 Gm. digested with 10 Cc. ammonia water, evaporated to dryness, taken up with nitric acid, and evaporated, dry residue,	Should not respond to Gutzeit's test.
<i>Precipitated sulphur.</i>		
<i>Boric acid.</i>	5 Cc. saturated aqueous solution,	Should not respond to Gutzeit's test.
<i>Hydrochloric acid.</i>	5 Cc. of a dilution (1 in 10),	Should not respond to Gutzeit's test.
<i>Diluted hydrobromic acid.</i>	5 Cc. of the official acid,	Should not respond to Gutzeit's test.
<i>Diluted hydriodic acid.</i>	5 Cc. of official acid, poured into 3 Cc. nitric acid in a beaker, 10 Cc. water added, and evaporated to dryness on water-bath, residue,	Should not respond to Gutzeit's test.
<i>Nitric acid.</i>	5 Cc. of a dilution (1 in 10),	Should not respond to Gutzeit's test.
<i>Sulphuric acid.</i>		
<i>Phosphoric acid.</i>	Prepared like diluted hydriodic acid, only 2 Cc. acid is used,	Should not respond to Gutzeit's test.
<i>Hypophosphorous acid.</i>		
<i>Potassium acetate.</i>	5 Cc. aqueous solution (1 in 10),	Should not respond to Gutzeit's test.
<i>Potassium sulphate.</i>		
<i>Sodium acetate.</i>		
<i>Sodium phosphate.</i>		
<i>Sodium pyrophosphate.</i>		
<i>Sodium sulphate.</i>		
<i>Magnesium sulphate.</i>		
<i>Glycerin.</i>	Prepared like diluted hydriodic acid, only 5 Cc. aqueous solution (1 in 10) is used,	Should not respond to Gutzeit's test.
<i>Potassium hypophosphite.</i>		
<i>Sodium hypophosphite.</i>		
<i>Calcium hypophosphite.</i>		
<i>Manganese hypophosphite.</i>		
<i>Methylene blue.</i>	Residue after igniting 2 Gm. chemical with sodium carbonate and potassium nitrate,	Should not respond to Gutzeit's test.
<i>Precipitated calcium phosphate.</i>	2 Gm. of dry chemical,	Should not respond to Gutzeit's test.
<i>Cerium oxalate.</i>	5 Cc. solution (1 in 10) in diluted hydrochloric acid,	Should not respond to Gutzeit's test.

The following chemicals tested by Gutzeit's method are prepared in a manner so intricate that the exact pharmacopœial wording is given.

Sodium Thiosulphate.—If to 5 Cc. of an aqueous solution of the salt (1 in 10), 3 Cc. of nitric acid be added, the solution cautiously evaporated to dryness on a water-bath, and the residue treated with distilled water, the liquid filtered, and the filtrate and washings evaporated to dryness, this residue should not respond to the Modified Gutzeit's Test for *arsenic*.

Reduced Iron.—To 0.5 Gm. of Reduced Iron, contained in a small covered beaker, add 20 Cc. of diluted sulphuric acid; after the reaction has somewhat subsided, warm the liquid on a water-bath until the reaction ceases, then collect any minute undissolved residue of impure iron arsenide upon a very small filter, rinse the beaker with water, add the rinsings to the filter, and wash the residue with water until free from acid reaction. Transfer the residue to the beaker by rinsing it back, and, after adding about 0.25 Gm. of potassium chlorate and 5 Cc. of hydrochloric acid, evaporate the solution slowly to dryness on a water-bath. Dissolve the residue in sufficient water to measure 50 Cc., then add 5 Cc. of this solution to 5 Cc. of a saturated solution of sulphurous acid, and heat the liquid on a water-bath for fifteen minutes, until all traces of sulphurous acid have been removed. The resulting solution should not respond to the Modified Gutzeit's test for *arsenic*.

Corrosive mercuric chloride.—
Mild mercurous chloride.—
Ammoniated mercury.—

} See p. 612.

Arsenic Test, Bettendorf's.—To a solution of the prescribed quantity of the substance to be tested in 5 Cc. of pure concentrated hydrochloric acid, contained in a clean test-tube, 5 Cc. of a saturated solution of freshly prepared stannous chloride in pure concentrated hydrochloric acid are added, and, after being heated for fifteen minutes while immersed in a bath of boiling water, the tube is allowed to stand for one hour. If arsenic be present in non-permissible amount, a brownish tint will become manifest when the tube is placed over a white surface and the solution viewed from above, comparison being made with a mixture of 5 Cc. each of pure concentrated hydrochloric acid and a saturated solution of stannous chloride, prepared under like conditions.

Note.—It is absolutely necessary that the solution of stannous chloride be freshly prepared, and that sulphates, sulphites, sulphides, and salts of mercury, gold, and selenium be absent from the reagents and chemicals being tested.

This is the method used in testing the following preparations:

CHEMICAL.	FORM TESTED.	
<i>Bismuth subnitrate.</i>	2 Gm. heated until converted into bismuth oxide,	Should not respond to Bettendorf's test.
<i>Bismuth subcarbonate.</i>	2 Gm. dry salt,	Should not respond to Bettendorf's test.
<i>Bismuth citrate.</i>	3 Gm. after ignition,	Should not respond to Bettendorf's test.
<i>Bismuth and ammonium citrate.</i>	3 Gm. after ignition,	Should not respond to Bettendorf's test.
<i>Bismuth subgallate.</i>	2 Gm. ignited, 5 Cc. nitric acid added, drop by drop, the solution evaporated to dryness and ignited; residue,	Should not respond to Bettendorf's test.
<i>Bismuth subsalicylate.</i>		
<i>Antimony and potassium tartrate.</i>	2 Gm. dissolved in 5 Cc. hydrochloric acid,	Should not respond to Bettendorf's test.

Zinc.—See p. 982.

Zinc Acetate.—See p. 982.

Zinc Bromide.—See p. 982.

Precipitated Zinc Carbonate.—See p. 982.

Zinc Chloride.—See p. 982.

Zinc Iodide.—See p. 982.

Zinc Oxide.—See p. 982.

Zinc Phenolsulphonate.—See p. 982.

Zinc Sulphate.—See p. 982.

Zinc Valerate.—See p. 983.

Calcium Chloride.—See p. 983.

Antimony and its compounds give, in acid solution with hydrogen sulphide, an orange precipitate of antimony sulphide, soluble in ammonium sulphide T.S. The following chemical is thus tested:

PRESENT AS INGREDIENT:

Antimony and Potassium Tartrate.—The aqueous solution of the salt possesses a slightly acid reaction, and yields with hydrochloric acid a white precipitate, soluble in an excess of the acid; but no precipitate occurs if tartaric acid has previously been added.

In a solution of Antimony and Potassium Tartrate, acidulated with hydrochloric acid, hydrogen sulphide T.S. produces an orange-red precipitate, which is soluble in ammonium sulphide T.S. and potassium hydroxide T.S.

Antimony and Potassium Tartrate is precipitated from its aqueous solution by tannic acid T.S., and yields a white precipitate with solutions of the alkaline carbonates and hydroxides, soluble in excess of the latter.

Sought as impurity in—

Zinc.—Hydrogen evolved on treatment of 1 Gm. with 20 Cc. diluted hydrochloric acid should not color strip of paper moistened with silver nitrate T.S.

Cadmium and its compounds give, in acid solution with hydrogen sulphide, a yellow precipitate, insoluble in ammonium sulphide T.S.

Sought as impurity in—

Zinc.—See p. 982.

Zinc Acetate.—See p. 982.

Zinc Bromide.—See p. 982.

Precipitated Zinc Carbonate.—See p. 982.

Zinc Chloride.—See p. 982.

Zinc Iodide.—See p. 982.

Zinc Oxide.—See p. 982.

Zinc Phenolsulphonate.—See p. 982.

Zinc Sulphate.—See p. 982.

Zinc Valerate.—See p. 983.

Copper salts give, with potassium ferrocyanide T.S., a red-brown precipitate of copper ferrocyanide. With ammonia water they give a blue-white precipitate of cupric hydroxide, which, when treated with excess of ammonia water, dissolves to an azure-blue liquid (see Cupric Ammonium Sulphate T.S., p. 972). If a bright piece of steel is dipped into a copper solution, it becomes coated with metallic copper. The following official chemicals are thus tested:

PRESENT AS INGREDIENT:

Copper Sulphate.—If a drop of the solution (1:20) be placed upon a bright piece of iron, a red film of metallic copper will be produced.

With potassium ferrocyanide T.S. the solution yields a deep reddish-brown precipitate.

If ammonia water be added to the solution, drop by drop, a pale blue precipitate of cupric hydroxide is formed, which redissolves in an excess of ammonia water, forming a deep azure-blue solution.

Sought as impurity in—

Bismuth Subnitrate.—

Bismuth Subcarbonate.—

Bismuth Subsalicylate.—

Bismuth Citrate.—

Bismuth and Ammonium Citrate.—

{ 3 Gm. dissolved in about 4 Cc. warm nitric acid, poured into 100 Cc. water, filtrate evaporated to 30 Cc. and again filtered, 5 Cc. of this filtrate should give no blue color with excess of ammonia water.

{ 3 Gm., when ignited and treated as in test for bismuth subnitrate just given, should give no blue color.

Bismuth Subgallate.—3 Gm. ignited, dissolved in nitric acid, added drop by drop, evaporated, ignited, redissolved in nitric acid, the solution concentrated to 4 Cc., and treated as in test for bismuth subnitrate given above, should give no blue color.

Solution of Formaldehyde.—See test for iron in solution of formaldehyde (p. 972).

Oil of Cajuput.—On shaking 5 Cc. of the Oil with 5 Cc. of water containing 1 drop of diluted hydrochloric acid, a reddish-brown color should not be produced in the acid liquid when separated from the Oil, if a drop of potassium ferrocyanide T.S. be added (absence of copper).

Sodium Chlorate.—See p. 982.

Zinc.—See p. 982.

Zinc Acetate.—See p. 982.

Zinc Bromide.—See p. 982.

Precipitated Zinc Carbonate.—See p. 982.

Zinc Chloride.—See p. 982.

Zinc Iodide.—See p. 982.

Zinc Oxide.—See p. 982.

Zinc Phenolsulphonate.—See p. 982.

Zinc Sulphate.—See p. 982.

Zinc Valerate.—See p. 983.

Sodium Arsenate.—See p. 983.

Lead Acetate.—See p. 983.

Lead Nitrate.—See p. 983.

Bismuth salts in concentrated acid solution yield, when poured into large amount of water, a white precipitate (of the "sub" salt of bismuth). **With hydrogen sulphide, solutions of bismuth salts give a black precipitate of bismuth sulphide.** The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Bismuth Subnitrate.—See p. 592.

Bismuth Subcarbonate.—If 3 Gm. of the salt be dissolved in just a sufficient quantity (about 4 Cc.) of warm nitric acid, and the solution poured into 100 Cc. of water, a white precipitate is produced.

Bismuth Citrate.—Like test for bismuth subcarbonate just given. Also: a solution of 1 Gm. of Bismuth Citrate in ammonia water, when treated with hydrogen sulphide in excess, yields a black precipitate.

Bismuth and Ammonium Citrate.—Like tests for bismuth citrate just given, except that the hydrogen sulphide test is performed in solution with sodium hydroxide.

Bismuth Subgallate.—0.1 Gm., agitated with hydrogen sulphide T.S., gives black precipitate.

Bismuth Subsalsicylate.—1 Gm., shaken with 10 Cc. diluted hydrochloric acid and the filtrate poured into excess of water, gives a heavy white precipitate of basic bismuth chloride.

Lead salts give, with hydrochloric acid, a white precipitate of lead chloride, which is sparingly soluble in water; with diluted sulphuric acid it gives a white precipitate of lead sulphate; with hydrogen sulphide, a black precipitate of lead sulphide; with potassium iodide, a yellow precipitate of lead iodide. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Lead Oxide.—See p. 599.

Lead Acetate.—The aqueous solution of Lead Acetate has a neutral or slightly alkaline reaction, and yields a black precipitate with hydrogen sulphide T.S., a yellow one with potassium iodide T.S., and a white one with diluted sulphuric acid.

Lead Nitrate.—Like test for lead acetate just given.

Sought as impurity in—

Bismuth Subnitrate.—3 Gm. salt is dissolved in 3 Cc. warm nitric acid, and solution is poured into 100 Cc. water. The filtrate is evaporated on water-bath to 30 Cc.,

and again filtered. Five Cc. of this filtrate mixed with an equal quantity of diluted sulphuric acid should not become cloudy.

Bismuth Subcarbonate.—Like test for bismuth subnitrate just given, only 4 Cc. nitric acid is used.

Bismuth Citrate.—
Bismuth and Ammonium Citrate.—

{ Like test for bismuth subnitrate just given, only the salt is ignited and dissolved in just a sufficient quantity of warm nitric acid.

Bismuth Subgallate.—
Bismuth Subsalsicylate.—

{ The salt is ignited, dissolved in just enough nitric acid, evaporated to dryness, and ignited, again dissolved in nitric acid and evaporated to 4 Cc. Rest of process as in test for bismuth subnitrate given above.

Silver Nitrate.—If 5 Cc. of an aqueous solution of the salt (1 in 10) be mixed with 20 Cc. of hot diluted sulphuric acid and heated to boiling, no turbidity should be perceptible, and upon standing no white precipitate should be deposited (absence of lead).

Sulphuric Acid.—On mixing the Acid carefully with 4 or 5 volumes of alcohol, no precipitate should be formed within one hour (absence of lead).

This test is purely physical, the lead salt not dissolving in alcohol.

Solution of Formaldehyde.—See test for iron in solution of formaldehyde (p. 972).

Sodium Chlorate.—See p. 982.

Zinc.—See p. 982.

Zinc Acetate.—See p. 982.

Zinc Bromide.—See p. 982.

Precipitated Zinc Carbonate.—See p. 982.

Zinc Chloride.—See p. 982.

Zinc Iodide.—See p. 982.

Zinc Oxide.—See p. 982.

Zinc Phenolsulphonate.—See p. 982.

Zinc Sulphate.—See p. 982.

Zinc Valerate.—See p. 983.

Sodium Arsenate.—See p. 983.

Calcium Chloride.—See p. 983.

Mercury Salts (both "ous" and "ic").—Give with hydrogen sulphide a black precipitate of mercuric sulphide.

Mercurous Salts.—Give with hydrochloric acid a white precipitate of mercurous chloride.

Sought as impurity in—

Solution of Mercuric Nitrate.—No precipitation or cloudiness should occur in the Solution on the addition of water, or of diluted hydrochloric acid (absence of mercurous salt).

Mercuric Salts.—Give no precipitate with hydrochloric acid. They give with ammonia a white precipitate (ammoniated mercury); with potassium iodide, a red precipitate of mercuric iodide, soluble in excess, and with potassium hydroxide, a yellow precipitate of mercuric oxide. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Ammoniated Mercury.—The solution of the salt in diluted nitric acid gives with potassium iodide T.S. a red precipitate.

Corrosive Mercuric Chloride.—See p. 612.

Silver Salts.—Give with hydrochloric acid, a curdy white precipitate of silver chloride, soluble in ammonia water, but insoluble in nitric acid. The following official chemicals are thus tested:

PRESENT AS INGREDIENTS:

Silver Nitrate.—See p. 622.

Mitigated Silver Nitrate.—An aqueous solution of Mitigated Silver Nitrate yields, with a slight excess of hydrochloric acid, a white precipitate, which is readily soluble in ammonia water.

Sought as impurity in—

Bismuth Subnitrate.—
Bismuth Subcarbonate.—
Bismuth Citrate.—
Bismuth and Ammonium Citrate.—
Bismuth Subgallate.—
Bismuth Subsalicylate.—

5 Cc. filtrate prepared exactly as outlined for the respective chemical in test for lead p. 977) should not “yield with hydrochloric acid a precipitate which is insoluble in a slight excess of the latter.”

Group Impurities.—Aside from the tests for specific impurities given on the foregoing pages, the pharmacopœia in several cases directs tests for groups of impurities, such as *heavy metals*, *excess of alkali*, and *non-volatile impurities*.

Time-limit Test for Heavy Metals.—This test is to be used to detect the presence of undesirable metallic impurities in official chemical substances or their solutions; these should not respond affirmatively within the stated time.

Ten Cc. of a solution of the substance in distilled water (1 in 20), contained in a test-tube of about 40-Cc. capacity, is acidulated with 1 Cc. of diluted hydrochloric acid (unless otherwise directed), warmed to about 50° C. (122° F.), and an equal volume of freshly prepared hydrogen sulphide T.S. added, and the mixture allowed to stand, in the well-stoppered test-tube, in a warm place, at 35° C. (95° F.), for at least half an hour. At the end of this time any coloration or turbidity is carefully noted, ammonia water is added in excess, and the solution again examined for a coloration or turbidity.

Before the addition of the ammonia water, the mixture should still possess the odor of hydrogen sulphide; if not, it should be thoroughly saturated with the gas and again set aside for half an hour.

Any change in the color of the solution which is being tested should be noted by comparison with the same volume of the hydrogen sulphide T.S. (which has been likewise acidulated), when viewed by reflected light while held against a white surface.

Antimony yields, upon the addition of hydrogen sulphide T.S. to highly diluted solutions, a pale yellow to orange color, or, to more concentrated solutions, an orange precipitate, which is soluble in test solutions of potassium hydroxide and ammonium sulphide, as well as in strong hydrochloric acid. The precipitate is insoluble in test solution of ammonium carbonate. The addition of ammonia water to the highly diluted hydrogen sulphide solution of antimony slightly intensifies the coloration.

Arsenic yields, upon the addition of hydrogen sulphide T.S. to highly diluted solutions, a pale yellow color, to more concentrated solutions, a yellow precipitate, which is soluble in test solutions of potassium hydroxide, ammonium sulphide, and ammonium carbonate, but is reprecipitated upon the addition of hydrochloric acid, insoluble in excess. The addition of ammonia water to the highly diluted hydrogen sulphide solution of arsenic slightly intensifies the yellow color.

Cadmium in very dilute solutions gives, with hydrogen sulphide T.S. or ammonium sulphide T.S., a pale yellow color; it yields in more concentrated solutions (not excessively acid) a yellow precipitate, which is insoluble in cold diluted hydrochloric acid, potassium hydroxide T.S., or ammonium sulphide T.S. (distinction from *arsenic*), and in solution of potassium cyanide. This precipitate is soluble in nitric and hydrochloric acids and in hot diluted hydrochloric and sulphuric acids.

Copper yields, upon the addition of hydrogen sulphide T.S. to highly diluted solutions, a pale brown color, or, to more concentrated solutions, a brownish-black precipitate, which is insoluble in diluted hydrochloric acid and test solution of potassium hydroxide, and is but very slightly soluble in test solution of ammonium sulphide. The precipitate is soluble in warm diluted nitric acid and also in solution of potassium or sodium cyanide. The addition of ammonia water to the highly diluted hydrogen sulphide solution containing copper slightly intensifies the coloration.

Iron.—Acidified solutions of ferrous iron do not react with hydrogen sulphide

T.S., but yield a dark coloration or black precipitate with ammonium sulphide T.S., or upon the addition of ammonia water to the hydrogen sulphide mixture. This latter precipitate is soluble in cold diluted hydrochloric acid.

Acidified solutions of ferric iron yield a white turbidity or precipitate of sulphur upon the addition of hydrogen sulphide T.S., but a dark coloration or black precipitate with ammonium sulphide T.S., or upon the addition of ammonia water to the hydrogen sulphide mixture. This precipitate is readily soluble in acetic and inorganic acids.

Lead yields, upon the addition of hydrogen sulphide T.S. or ammonium sulphide T.S. to highly diluted solutions, a pale brown coloration, or, to more concentrated solutions, a black precipitate, which is insoluble in diluted hydrochloric acid, and also in test solutions of potassium hydroxide and ammonium sulphide.

Zinc yields, with ammonium sulphide T.S. and with hydrogen sulphide T.S., either in neutral solution, or after acidulation with acetic acid (in the absence of free mineral acids), a white turbidity in highly diluted solutions, but with concentrated solutions a white precipitate, soluble in hydrochloric acid and insoluble in acetic acid.

Below are tabulated the official chemicals thus tested, with outline of method of preparation before applying the test:

CHEMICAL.	PREPARATION.
<i>Solution of hydrogen dioxide.</i>	1 Cc. solution is mixed with 1 Cc. ammonia water evaporated to dryness; this residue, dissolved in 10 Cc. distilled water, containing 1 Cc. diluted hydrochloric acid, should not respond to test.
<i>Boric acid.</i>	Aqueous solution (1 in 25) should not respond to test.
<i>Hydrochloric acid.</i>	} Aqueous dilution (1 in 20) should not respond to test.
<i>Acetic acid.</i>	
<i>Diluted hydrobromic acid.</i>	} 10 Cc. of diluted acid should not respond to test.
<i>Diluted hydriodic acid.</i>	
<i>Nitric acid.</i>	Neutralized with ammonia water and diluted to 1 in 20 should not respond to test.
<i>Sulphuric acid.</i>	Aqueous dilution (1 in 20) should not respond to test.
<i>Phosphoric acid.</i>	10 Cc. aqueous dilution (1 in 20) should not respond to test.
<i>Hypophosphorous acid.</i>	10 Cc. official acid should not respond to test.
<i>Potassium hydroxide.</i>	} Aqueous solution (1 in 20), slightly acidulated with hydrochloric acid, should not respond to test.
<i>Potassium bicarbonate.</i>	
<i>Potassium bromide.</i>	
<i>Potassium carbonate.</i>	
<i>Potassium and sodium tartrate.</i>	
<i>Potassium iodide.</i>	
<i>Potassium nitrate.</i>	
<i>Potassium sulphate.</i>	
<i>Sodium hydroxide.</i>	
<i>Sodium borate.</i>	
<i>Sodium bromide.</i>	
<i>Sodium carbonate.</i>	
<i>Sodium bicarbonate.</i>	
<i>Sodium chloride.</i>	
<i>Sodium iodide.</i>	
<i>Sodium nitrate.</i>	
<i>Sodium phenolsulphonate.</i>	
<i>Sodium phosphate.</i>	
<i>Sodium pyrophosphate.</i>	
<i>Sodium sulphate.</i>	
<i>Ammonium bromide.</i>	
<i>Ammonium carbonate.</i>	
<i>Ammonium iodide.</i>	
<i>Calcium bromide.</i>	
<i>Strontium bromide.</i>	
<i>Strontium iodide.</i>	
<i>Glycerin.</i>	

CHEMICAL.	PREPARATION.
<i>Potassium hypophosphite.</i> <i>Sodium hypophosphite.</i> <i>Lithium citrate.</i> <i>Calcium hypophosphite.</i>	Aqueous solution (1 in 20), acidulated with hydrochloric acid, should not respond to test.
<i>Potassium bitartrate.</i>	
<i>Sodium benzoate.</i> <i>Sodium salicylate.</i> <i>Lithium benzoate.</i> <i>Lithium salicylate.</i> <i>Ammonium benzoate.</i> <i>Ammonium salicylate.</i> <i>Ammonium valerate.</i> <i>Strontium salicylate.</i>	Aqueous solution (1 in 20), acidulated with hydrochloric acid and filtered; the filtrate should not respond to test.
<i>Ammonia water.</i>	
<i>Magnesium oxide.</i> <i>Magnesium carbonate.</i> <i>Cerium oxalate.</i> <i>Aluminum hydroxide.</i>	Solution (1 in 20) in diluted hydrochloric acid should not respond to test.
<i>Lithium carbonate.</i> <i>Precipitated calcium carbonate.</i> <i>Precipitated calcium phosphate.</i>	
<i>Manganese sulphate.</i>	Aqueous solution (1 in 20), to which a few drops hydrochloric acid and of chlorine water is added, should not respond to test.
<i>Potassium acetate.</i> <i>Potassium citrate.</i> <i>Sodium acetate.</i> <i>Sodium citrate.</i>	Aqueous solution (1 in 20), slightly acidulated with acetic acid, should not respond to test.
<i>Sodium bisulphite.</i> <i>Sodium nitrite.</i> <i>Sodium sulphite.</i>	
<i>Lithium bromide.</i> <i>Ammonium chloride.</i> <i>Alum.</i> <i>Magnesium sulphate.</i>	Aqueous solution (1 in 20) should not respond to test.
<i>Aluminum sulphate.</i> <i>Sodium thiosulphate.</i>	
<i>Lactic acid.</i>	10 Cc. aqueous solution (1 in 10) should not respond to test.
<i>Ferrous sulphate.</i>	If 1 Gm. of the salt be dissolved in about 25 Cc. of water containing 1 Cc. of diluted sulphuric acid, the solution heated to boiling, oxidized with nitric acid, and then mixed with a slight excess of ammonia water, the filtrate from the reddish-brown precipitate produced should be colorless, and, after acidulating with hydrochloric acid, should not respond to test.
<i>Ferric chloride.</i>	If 1 Gm. of the salt, dissolved in 25 Cc. of boiling water, be treated with an excess of ammonia water, the filtrate should be colorless, and, after acidulation with hydrochloric acid, 20 Cc. of the solution should not respond to test.

CHEMICAL.	PREPARATION.
<i>Ferric hypophosphite.</i>	If 1 Gm. of the salt be dissolved in about 25 Cc. of boiling water by the aid of sufficient hydrochloric acid, added drop by drop, then a slight excess of ammonia water added, the filtrate from the reddish-brown precipitate should be colorless, and, after acidulating with hydrochloric acid, should not respond to test.
<i>Antimony and potassium tartrate.</i>	If to the aqueous solution of the salt (1 in 20) just sufficient sodium hydroxide T.S. be added to redissolve the precipitate formed, and if to this solution an equal volume of freshly prepared hydrogen sulphide T.S. be added, no coloration should be noticeable after standing in a warm place for half an hour when viewed by reflected light while held against a white surface (absence of <i>heavy metals</i>).
<i>Copper sulphate.</i>	If the aqueous solution of the salt (1 in 20) be heated to boiling with an excess of sodium hydroxide T.S., until all of the copper has been converted into black cupric oxide, it will yield a colorless filtrate, which, after acidulation with acetic acid, should not respond to test.
<i>Potassium chlorate.</i>	The aqueous solution of the salt (1 in 20) should not become discolored upon the addition of ammonium sulphide T.S. (absence of <i>heavy metals</i>).
<i>Citric acid.</i>	The aqueous solution (1 in 20), mixed with a few drops of hydrochloric acid, should not respond to the Time-limit Test for <i>heavy metals</i> , nor, upon the subsequent addition of ammonia water in slight excess, to the solution, should the liquid acquire more than a faint tint (limit of <i>iron</i>).
<i>Tartaric acid.</i>	Like test for citric acid just given, except ammonia water is omitted.

Modifications of the Heavy Metal Test:

LEAD AND COPPER SOUGHT IN—

Sodium Chlorate.—The aqueous solution of the salt (1 in 20), upon the addition of 1 Cc. of ammonium sulphide T.S., should not develop a dark coloration (absence of *lead*, *copper*, etc.).

ARSENIC, CADMIUM, LEAD, AND COPPER SOUGHT IN—

Zinc.—If 1 Gm. of Zinc be dissolved in a mixture of 10 Cc. each of nitrohydrochloric acid and water, the solution evaporated to dryness, the residue moistened with 2 Cc. of hydrochloric acid, and again evaporated, and the final residue dissolved in 10 Cc. of water, this solution should not respond to the Time-limit Test for *arsenic*, *cadmium*, *lead*, and *copper* (see p. 979); in applying this test the addition of ammonia water should be omitted.

Zinc Acetate.—Ten Cc. of the aqueous solution of the salt (1 in 20), to which 1 Cc. of hydrochloric acid has been added, should not respond to the Time-limit Test for *arsenic*, *cadmium*, *lead*, and *copper* (see p. 979); in applying this test the addition of ammonia water should be omitted.

Zinc Bromide.—Like test for zinc acetate just given.

Precipitated Zinc Carbonate.—1.25 Gm. salt is dissolved in 10 Cc. diluted sulphuric acid and 10 Cc. water, the filtrate, acidulated with hydrochloric acid, should not respond to the Time-Limit Test for *arsenic*, *cadmium*, *lead*, and *copper* (see p. 979); in applying this test the addition of ammonia water should be omitted.

Zinc Chloride.— } Like test for zinc acetate given above.

Zinc Iodide.— }

Zinc Oxide.—1 Gm. salt digested with 10 Cc. diluted hydrochloric acid and 10 Cc. water, and the filtrate, acidulated with hydrochloric acid, should not respond to the test for *arsenic*, *cadmium*, *lead*, and *copper* (p. 979), omitting ammonia water.

Zinc Phenolsulphonate.— } Like test for zinc acetate given above.

Zinc Sulphate.— }

Zinc Valerate.—0.5 Gm. salt dissolved in mixture of 0.5 Cc. hydrochloric acid and 9 Cc. water. After filtering through a small wetted filter, the clear solution should not respond to the Time-limit Test for *arsenic*, *cadmium*, *lead*, and *copper* (p. 979); in applying this test the addition of ammonia water should be omitted.

LEAD, COPPER, AND IRON SOUGHT IN—

Sodium Arsenate.—If to 5 Cc. of an aqueous solution (1 in 20) 1 Cc. of ammonium sulphide T.S. be added, no dark coloration should appear (absence of *lead*, *copper*, *iron*, etc.).

ARSENIC AND LEAD SOUGHT IN—

Calcium Chloride.—The aqueous solution of the salt (1 in 20), slightly acidulated with hydrochloric acid, should not respond to the Time-limit Test for *arsenic* or *lead*.

IRON AND ALUMINUM SOUGHT IN—

Copper Sulphate.—If hydrogen sulphide gas be passed through 10 Cc. of the aqueous solution of the salt (1 in 20), to which 1 Cc. of diluted hydrochloric acid has been added, until all of the copper is precipitated as sulphide, one-half of the colorless filtrate should not be colored or rendered turbid upon the addition of ammonia water, nor should the remaining portion of the filtrate yield, upon evaporation, a weighable residue (limit of *iron*, *aluminum*, etc.).

IRON AND COPPER SOUGHT IN—

Lead Acetate.—Aqueous solution (1 in 10) gives pure white precipitate with potassium ferrocyanide T.S.

Lead Nitrate.—Like test for lead acetate just given.

ZINC AND IRON SOUGHT IN—

Lead Acetate.—If to the aqueous solution of the salt (1 in 20) hydrochloric acid be added until no further precipitate is produced, the remainder of the lead removed from the filtrate by hydrogen sulphide, and the liquid filtered, a portion of the second filtrate should not be affected by the addition of a slight excess of ammonia water (absence of *zinc* and *iron*).

Lead Nitrate.—Like test for lead acetate just given.

Free Alkali Tests:

Potassium Iodide.—If 1 Gm. of the salt be dissolved in 10 Cc. of water, and 0.1 Cc. of tenth-normal sulphuric acid V.S. be added, no color should be produced by the subsequent addition of a drop of phenolphthalein T.S., even after heating (limit of *alkali*).

Sodium Iodide.—Like test for potassium iodide just given, except amount of water is omitted.

Sodium Bromide.—Like test for potassium iodide just given, except for use of words "even after boiling."

Sodium Hypophosphite.—The aqueous solution (1 in 20) should not be colored red by the addition of a drop of phenolphthalein T.S. nor effervesce on the addition of an acid (absence of *caustic alkali* and *carbonate*).

Sodium Thiosulphate.—Aqueous solution (1 in 20) not colored red by drop of phenolphthalein T.S.

Lithium Benzoate.—If excess of hydrochloric acid be added to a concentrated aqueous solution of 0.6 Gm. salt, benzoic acid is precipitated. If the filtrate and washings from this precipitate be evaporated almost to dryness on a water-bath, in a flat-bottomed flask of 50-Cc. capacity, and 10 Cc. of amyl alcohol (boiling-point 132° C.) be added, and the mixture cautiously heated until the lower aqueous layer has evaporated, then, after the addition of 3 drops of hydrochloric acid and boiling for three minutes, the resulting insoluble residue should weigh not more than 0.004 Gm. (limit of *other alkalis*). The removal of the water from the amyl alcohol mixture is facilitated by passing a current of air through the hot solution.

Lithium Bromide.—Like test for lithium benzoate just given, except 0.4 Gm. salt should yield not more than 0.004 Gm., insoluble in amyl alcohol.

Solution of Lime.—See test for carbonates in solution of lime (p. 969).

Precipitated Zinc Carbonate.—If 1 Gm. of the salt be placed in a flask with 10 Cc. of boiling water, and 2 drops of phenolphthalein T.S. added, not more than 1 Cc. of tenth-normal hydrochloric acid V.S. should be required to discharge the red color (limit of *alkali*).

Zinc Oxide.—Like test for precipitated zinc carbonate just given.

Thymol Iodide.—If 0.5 Gm. be shaken with 10 Cc. of water, and the mixture filtered, no blue color should be imparted by the filtrate to red litmus-paper (absence of *alkalis*).

Non-volatile Impurity Tests:

Diluted Hydrobromic Acid.—If 10 Cc. of Diluted Hydrobromic Acid be evaporated to dryness and heated to 110° C. (230° F.), no appreciable residue should remain (limit of *non-volatile impurities*).

Hydrochloric Acid.—Like test for diluted hydrobromic acid just given, except use of words "evaporated in a platinum or porcelain dish."

Nitric Acid.—Like test for diluted hydrobromic acid given above.

Sulphuric Acid.—Supersaturated with ammonia water, it leaves no appreciable residue on evaporation and ignition.

SYSTEMATIC CHEMICAL ANALYSIS

The pharmacopœial tests given on preceding pages, while important and valuable, give us information that is somewhat disjointed.

If we suspect that a liquid contains zinc, we may try the tests for zinc given on p. 969; but suppose the liquid contains magnesium and iron as well as zinc, how would we then proceed? Or suppose we had no idea what metals the solution contains, how would we begin?

In analytic chemistry, as in everything else in science, system is the secret of success, and it is to systematic analysis to which we now must refer. This is performed by separating the several elements in a solution into groups by the use of certain chemicals which we call "group reagents." The important group reagents are:

First, hydrochloric acid (HCl); second, hydrogen sulphide (H_2S); third, ammonium sulphide (NH_4HS); fourth, ammonium carbonate, $(NH_4)_2CO_3$; applying them one after another to the solution under examination.

Now, suppose we have a solution containing *all* the elements used in pharmacy, we would take the solution and add

Hydrochloric acid, when there would be precipitated out Hg (ous), Pb, and Ag as insoluble chlorides. The liquid is separated from the precipitate by filtration, and through the filtrate we would pass

Hydrogen sulphide (gas), when we would get a precipitate of: (a) Cd, Cu, Hg (ic), Pb, Bi. (b) As, Sb, and Sn as insoluble sulphides. The precipitate can be separated into groups "a" and "b" by digestion in NH_4HS , in which group "b" dissolves, leaving group "a" behind. In the meanwhile the filtrate from the H_2S precipitate is treated with NH_4Cl , NH_4OH , and

Ammonium sulphide, which precipitates from it (a) Fe, Al, Cr; (b) Zn, Mn, Co, and Ni. All save those in italics as sulphides. The italicized as hydrates.

Group "a" of this precipitate, when dissolved in hydrochloric and nitric acid, is precipitated by ammonia water. Group "b," under

same treatment, is not. By this method the two groups are separated. The filtrate from the NH_4HS precipitate is now treated with

Ammonium carbonate, when down comes Ba, Sr, and Ca, as insoluble carbonates. To the filtrate from this precipitate is added

Ammonium phosphate, when there is precipitated Mg as phosphate. The filtrate will now hold only Li, Na, K, and NH_4 , and this is considered the last group.

By this method we have separated the metals into seven groups, as follows:

I.	Li	K	Na	NH_4
II.	Mg	Ca	Sr	Ba
III.	Zn	Mn	Co	Ni
IV.	Al	Fe	and Cr	
V.	As	Sb	Sn	
VI.	Cd	Cu	Hg	(ic) Bi
VII.	Hg	(ous)	Pb	and Ag.

Only the symbols are given above, as it is expected that the preceding chapters of this book have been read and mastered.

Some books on analytic chemistry number these groups differently, beginning with Hg (ous) and Ag as first group, but this is of small importance, the main point being the separation of the twenty-four metals into seven simple groups, from which, in turn, the metals themselves can be separated without great difficulty. How this separation of the small groups is carried on is beyond the scope of this book, and the reader is referred to one of the books on analytic chemistry mentioned in Preface.

A summary of the more important individual tests may be useful. The following metals give characteristic color with *borax bead*:

Manganese.....	Amethyst.
Cobalt.....	Blue.
Copper.....	Green.
Iron.....	Red.

The following flame tests are characteristic:

Strontium and lithium.....	Crimson.
Calcium.....	Red-yellow.
Copper.....	Blue-green.
Potassium.....	Violet.
Sodium.....	Yellow.
Boric acid and alcohol.....	Green.

The following hydrogen sulphide precipitates are characteristic:

White.....	Zinc (from alkaline solution).
Flesh color.....	Manganese (from alkaline solution).
Yellow.....	Arsenic and tin (from acid solution).
Orange.....	Antimony (from acid solution).
Blacks.....	Mercury, copper, lead (from acid solution).
The other elements will not precipitate with H_2S .	

The following special tests are characteristic:

SUBSTANCE.	REAGENT.	REACTION.
<i>Acids and their salts.</i>		
H ₂ SO ₄ .	Barium chloride.	White precipitate.
HCl.	Silver nitrate.	White precipitate.
HBr.	Silver nitrate.	Yellow precipitate.
Phosphoric acid.	Magnesium sulphate.	White crystalline precipitate.
Carbonic acid.	Lime water.	White precipitate.
Nitric acid.	Green FeSO ₄ solution.	Changed to <i>red</i> or <i>black</i> Fe ₂ (SO ₄) ₃ solution.
Thiosulphuric acid.	Sulphuric acid.	Vapors of SO ₂ and precipitate of sulphur.
Sulphurous acid.	Sulphuric acid.	Vapors of SO ₂ .
Tartaric acid.	Silver nitrate.	Mirror of metallic silver.
Acetic acid.	H ₂ SO ₄ and alcohol.	Odor of acetic ether.
<i>Metallic Salts.</i>		
Zinc.	Hydrogen sulphide.	White precipitate.
Calcium.	Ammonium oxalate.	White precipitate.
Magnesium.	Sodium phosphate.	White precipitate.
Iron (ic).	Potassium ferrocyanide.	Blue precipitate.
Iron (ous).	Potassium ferricyanide.	Blue precipitate.
Silver.	Sodium chloride.	White precipitate.
Aluminum.	Solution of soda.	White gelatinous precipitate.
Lead.	Potassium chromate.	Yellow precipitate.
Arsenic.	} Test by Marsh's, Fleitmann's, or Gutzeit's test, as explained on p. 573.	
Antimony.		

Tests for organic chemicals are too numerous and varied to admit of a tabulated statement; hence the reader is referred to the pharmacopœial tests for such substances, found scattered through Part IV.

CHAPTER LIV

QUANTITATIVE ANALYSIS

INORGANIC VOLUMETRIC ANALYSIS

As mentioned on p. 944, we are able to examine chemicals not merely as to ingredients, but also can ascertain the quantity of each ingredient. The latter work is the object of quantitative analysis. The three methods of quantitative analysis usually employed are volumetric analysis, gravimetric analysis, and gasometric analysis.

VOLUMETRIC ANALYSIS

is the most largely employed process of quantitative analysis as far as pharmacy is concerned; the pharmacopœia directing this method in the estimation of the strength of drugs whenever possible. The volumetric processes of the pharmacopœia, with but two exceptions (the use of Fehling's solution and the use of Koppeschaar's solution, p. 1047), fall into four groups:

I. Acidimetry or alkalimetry.

II. The use of iodine volumetric solution or its complement, sodium thiosulphate volumetric solution.

III. Oxidation with potassium permanganate volumetric solution or with potassium dichromate volumetric solution.

IV. The use of silver nitrate volumetric solution or its complements, potassium sulphocyanate volumetric solution and sodium chloride volumetric solution.

Before discussing these several groups of volumetric analysis it is advisable to quote what the pharmacopœia says about volumetric solutions, with special emphasis laid on the explanation of *normal solutions*, *decinormal solutions*, and *centinormal solutions*.

VOLUMETRIC SOLUTIONS

NOTE.—It is absolutely necessary that the measuring vessels employed in the operations of volumetric analysis, consisting of burettes, flasks, mixing cylinders, pipettes, etc., should agree among themselves accurately in their graduation at the standard temperature selected. It is immaterial what standard temperature has been selected for the graduation of the vessels.

All volumetric solutions must be prepared at the standard temperature of 25° C. (77° F.), and the solutions must be used in the titrations at a temperature not below 21° C. (69.8° F.), nor above 29° C. (84.2° F.).

All bottles in which volumetric solutions are to be kept, as well as the burettes or pipettes in which they are to be measured, should, prior to use, be thoroughly rinsed with distilled water, then with two or three small portions of the solution that they are to contain. When not in use, burettes should be kept filled with distilled water.

Normal volumetric solutions ($\frac{N}{1}$) are those which contain in one liter, in any stated reaction, the chemical equivalent of one gramme of hydrogen. If the molecule of the reagent is univalent, one liter will contain the weight in grammes equal to the molecular weight of the reagent; if bivalent, a weight in grammes equal to one-half its molecular weight; if trivalent, a weight in grammes equal to one-third its molecular weight.

Thus, hydrochloric acid, $\text{HCl} = 36.18$, having but one H atom replaceable by a basic element, has 36.18 Gm. of absolute HCl in 1000 Cc. of the normal volumetric solution; while sulphuric acid, $\text{H}_2\text{SO}_4 = 97.35$, having two replaceable H atoms, contains only one-half this number, or 48.675 grammes of absolute H_2SO_4 in 1000 Cc. of its normal solution. Potassium hydroxide, $\text{KOH} = 55.74$, has but one K to replace one H in acids, hence its normal solution contains 55.74 grammes of pure KOH in one liter. Again, one molecule of potassium dichromate in oxidation liberates *three* atoms of oxygen, which are capable of oxidizing *six* atoms of ferrous to ferric iron. Therefore, each molecule of the dichromate, yielding *three* atoms of oxygen, is equivalent to *six* atoms of hydrogen. Hence, the normal solution should contain $\frac{292.28}{6}$ or 48.713 Gm. in 1000 Cc. Two molecules of potassium permanganate, $2\text{KMnO}_4 = 313.96$, in oxidation, give off five atoms of O, which are equivalent to ten atoms of H; hence its normal solution should contain $\frac{313.96}{10}$, or 31.396 Gm. in 1000 Cc.

Solutions containing in 1000 Cc. one-tenth of the quantity of the active reagent in the normal solution are called tenth-normal ($\frac{N}{10}$); those containing one-hundredth, hundredth-normal ($\frac{N}{100}$); one-fiftieth, fiftieth-normal ($\frac{N}{50}$); those containing twice the amount, double-normal ($\frac{2N}{1}$); half the amount, half-normal ($\frac{N}{2}$).

Solutions containing quantities of the active reagent having *no* simple relation to the molecular weight are called empirical.

In the majority of the assays it has been directed that an integral quantity (1, 2, 5, or 10 Gm.) of the substance which is to be tested be weighed, dissolved in water, and made up to a definite volume, then an aliquot portion (representing the fractional weight of the substance required) is measured off for titration. Fractional parts of a Cc. may be measured by means of a 1 Cc. pipette graduated in one-tenth or one-twentieth divisions.

Acidimetry and Alkalimetry.—This group includes the estimation of alkalis by use of a volumetric solution of an acid or the estimation

of acids by the use of a volumetric solution of an alkali. The process can be best illustrated by a typical example. Taking the pharmacopœial estimation of potassium hydroxide, for instance, we find (p. 998) that it is accomplished by use of normal volumetric solution of sulphuric acid, and the theory of this kind of estimation is that sufficient of the acid solution is added to make the product exactly neutral—to make pure potassium sulphate. To know when this neutrality is reached, we use some substance which by change of color will show whether the liquid is acid or alkaline. Litmus can be used for this purpose, for we learned long since that litmus turns red with acids and blue with alkalis. But litmus is not exact enough. We need some substances which will change their tint when there is but a fraction of 1 per cent. of free acid or free alkali present. Such a coloring-matter is called an *indicator*, and chief among those used in this class of volumetric analysis are the following:

Phenolphthalein, red with alkalis, colorless with acids.

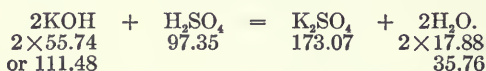
Methyl orange, yellow with alkalis, red with acids.

Hematoxylin, violet with alkalis, yellow with acids.

Iodeosin, pink with alkalis, colorless with acids.

Turning to the process, suppose we combine potassium hydroxide with sulphuric acid to a neutral salt. Absolutely pure potassium hydroxide is KOH; molecular weight, 55.74. Absolutely pure sulphuric acid is H₂SO₄; molecular weight, 97.35. The molecular weights just given are estimated from the "exact" atomic weights given by the pharmacopœia and not from the "round number" atomic weights found in elemental table on p. 367.

The reaction between potassium hydroxide and sulphuric acid can be expressed by the following equation:



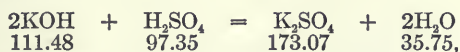
We see from the equation that absolute potassium hydroxide combines with absolute sulphuric acid in the proportion of 111.48 Gm. of the hydroxide to 97.35 Gm. of the acid, and were we to prepare solutions of these two bodies of such strength that equal volumes of the two solutions would exactly neutralize each other, the solutions would have to be prepared to the ratio of 111.48 to 97.35.

To make it plainer: were we to prepare a liter of solution containing exactly 111.48 Gm. absolute potassium hydroxide, a liter of solution of sulphuric acid to neutralize it would have to hold 97.35 Gm. of absolute acid; or if we prepared a solution of potassium hydroxide containing in one liter 55.74 Gm. of absolute hydroxide ($\frac{1}{2}$ of 111.48), then a liter of solution of sulphuric acid that would neutralize the hydroxide must contain one-half of 97.35 or 48.675 Gm. of absolute sulphuric acid, and such is the strength of normal sulphuric acid, as reference to p. 990 will show. The foundation to acidimetric work is the strength of normal potassium hydroxide solution and normal sodium hydroxide solution, each of which represents in each liter of solution the number of grammes of absolute sodium hydroxide equal to the molecular weight of the substance employed. Thus, normal potassium hy-

dioxide solution contains 55.74 Gm. of hydroxide to the liter, and normal sodium hydroxide contains 39.76 Gm. of hydroxide to the liter. On the other hand, the dibasic acids—sulphuric acid and oxalic acid—are made into normal volumetric solutions by using to a liter of solution, the number of grammes of acid corresponding to *half* the molecular weight, since it takes only half a molecule (if the phrase is permissible) of either of these acids to neutralize one molecule of either of the two alkalis.

The use of a volumetric solution in testing is simple. The volumetric solution is poured into a graduated tube called a burette (see Fig. 230, p. 945), while the substance to be examined, after careful weighing, is dissolved and so placed that the contents of the burette can be gradually run into it. The indicator—usually phenolphthalein—is poured into the matter to be examined. Let us suppose this substance is an alkali: then the solution will be colored purple red. Into this the volumetric solution is added gradually and stopped the moment that a drop of the reagent discharges the color of the solution, which will mean that the solution is no longer alkaline. Then a simple reckoning—amount of volumetric solution in burette *before*, minus amount *after*, dropping into tested solution, will give us the number of cubic centimeters of volumetric solution used in neutralizing the tested substance, and from these figures we can, by aid of the pharmacopœia, estimate strength of the tested substance.

Applying these directions to the estimation of potassium hydroxide, suppose we weigh out 1 Gm. of the chemical, dissolve it in water, and titrate it; treat it with normal sulphuric acid V.S. (V.S. meaning volumetric solution). We find the gramme of potassium hydroxide takes 16 Cc. normal sulphuric acid (abbreviated nH_2SO_4) to neutralize it. From the equation—



we see that 97.35 Gm. absolute $\text{H}_2\text{SO}_4 = 111.48$ Gm. absolute KOH. We have learned that a liter of $\text{nH}_2\text{SO}_4 = \frac{97.35}{2}$ Gm., or 48.67 Gm. absolute H_2SO_4 . Since 48.67 Gm. absolute $\text{H}_2\text{SO}_4 = \frac{111.48}{2}$ Gm., or 55.74 Gm. absolute KOH, we find that

1000 Cc. $\text{nH}_2\text{SO}_4 = 55.74$ Gm. absolute KOH.

1 Cc. $\text{nH}_2\text{SO}_4 = 0.05574$ Gm. absolute KOH.

Hence 16 Cc. $\text{nH}_2\text{SO}_4 = 16 \times 0.05574$ Gm. absolute KOH.

0.89224 Gm. absolute KOH.

Hence 1 Gm. potassium hydroxide we examined contained 0.89224 Gm. absolute KOH, and was therefore 89 per cent. absolute.

Reference to p. 990 will show that the factor "1 Cc. $\text{nH}_2\text{SO}_4 = 0.05574$ Gm. absolute KOH" is found under the table of values of 1 Cc. normal sulphuric acid V.S., and it need here be said that each particular value of 1 Cc. normal acid found therein has been worked out by process of reasoning just given. The same obtains with the equivalent tables found under the several volumetric solutions, and incidentally it might be noted that one cubic centimeter of a normal acid or of normal alkali is represented as the same number of milligrammes of the substance tested as the molecular weight of the substance if it be mono-

basic or monacid, half the molecular weight if it be dibasic or diacid, one-third the molecular weight if it be tribasic or triacid. The following volumetric solutions are used in acidimetry and alkalimetry:

NORMAL SULPHURIC ACID VOLUMETRIC SOLUTION

$\text{H}_2\text{SO}_4 = 97.35.$

48.675 Gm. in 1000 Cc.

Carefully mix 30 Cc. of pure, concentrated sulphuric acid of specific gravity 1.826 at 25° C. (77° F.) with sufficient water to measure about 1050 Cc., and allow the liquid to cool to 25° C. (77° F.). Measure from a burette 10 Cc. of this liquid (which is yet too concentrated) into a flask, add 2 drops of methyl-orange T.S., and afterward, from a burette, a freshly standardized normal potassium hydroxide V.S., shaking after each addition, and, toward the end of the operation, reducing the flow to drops, until the red tint of the solution changes to a permanent pale yellow after thorough shaking. Note the number of Cc. of normal potassium hydroxide V.S. consumed. Then dilute the sulphuric acid solution so that equal volumes of this and of normal potassium hydroxide V.S., at 25° C. (77° F.), exactly neutralize each other.

EXAMPLE.—Assuming that 10 Cc. of the acid solution first prepared had required exactly 11.2 Cc. of normal potassium hydroxide V.S., each 10 Cc. of the former must be diluted to 11.2 Cc., or each 1000 Cc. to 1120 Cc.

After the liquid is thus diluted, a new trial should be made in the manner above described, in which 50 Cc. of the acid solution should require for neutralization exactly 50 Cc. of normal potassium hydroxide V.S., at 25° C. (77° F.). If necessary, a new adjustment should be made to render the correspondence perfect.

One Cubic Centimeter of Normal Sulphuric Acid V.S. is the equivalent of:

	Gramme.
Sulphuric Acid, absolute, H_2SO_4	0.048675
Ammonia Gas, NH_3	0.01693
Ammonium Carbonate, $(\text{NH}_4)_2\text{CO}_3$	0.047705
Ammonium Carbonate [U. S. P.], $\text{NH}_4\text{HCO}_3 \cdot \text{NH}_4\text{NH}_2\text{CO}_2$	0.052003
Calcium Hydroxide, $\text{Ca}(\text{OH})_2$	0.03678
Lead Acetate, crystallized, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$	0.188075
Lead Subacetate, assumed as $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$	0.135935
Lithium Carbonate, Li_2CO_3	0.036755
Magnesium Carbonate, $(\text{MgCO}_3)_4 \cdot \text{Mg}(\text{OH})_2 + 5\text{H}_2\text{O}$	0.048226
Magnesium Oxide, MgO	0.02003
Potassium Acetate, $\text{KC}_2\text{H}_3\text{O}_2$, (after ignition).....	0.09744
Potassium Bicarbonate, KHCO_3	0.09941
Potassium Bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$, (after ignition).....	0.18678
Potassium Carbonate, anhydrous, K_2CO_3	0.068635
Potassium Citrate, cryst., $\text{K}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$ (after ignition).....	0.10736
Potassium Hydroxide, KOH	0.05574
Potassium and Sodium Tartrate, $\text{KNaC}_4\text{H}_4\text{O}_6 + 4\text{H}_2\text{O}$ (after ignition).....	0.14009
Sodium Acetate, $\text{NaC}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$ (after ignition).....	0.13510
Sodium Benzoate, $\text{NaC}_7\text{H}_5\text{O}_2$ (after ignition).....	0.14301
Sodium Bicarbonate, NaHCO_3	0.08343
Sodium Borate, crystallized, $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$	0.18966
Sodium Hydroxide, NaOH	0.03976
Sodium Salicylate, $\text{NaC}_7\text{H}_5\text{O}_3$ (after ignition).....	0.15889
Zinc Oxide, ZnO	0.04039

HALF-NORMAL SULPHURIC ACID VOLUMETRIC SOLUTION

$\text{H}_2\text{SO}_4 = 97.35.$

24.3375 Gm. in 1000 Cc.

Dilute 500 Cc. of normal sulphuric acid with sufficient water to measure 1000 Cc. at 25° C. (77° F.).

This standard solution is chiefly employed in the titration of the organic salts of sodium and potassium in conjunction with methyl-orange as indicator. For this purpose a special experiment should be made in which an accurately measured volume of 10 Cc. of normal potassium hydroxide V.S. should, after adding 2 drops of methyl-orange T.S., require exactly 20 Cc. of the half-normal sulphuric acid V.S. for neutralization.

If necessary, an adjustment should be made to render the correspondence perfect.

TENTH-NORMAL SULPHURIC ACID VOLUMETRIC SOLUTION

 $\text{H}_2\text{SO}_4 = 97.35.$

4.8675 Gm. in 1000 Cc.

Dilute 100 Cc. of normal sulphuric acid with sufficient water to measure 1000 Cc. at 25°C . (77°F .).

This standard solution is employed in conjunction with the fiftieth-normal potassium hydroxide V.S. in the titration of alkaloids, with hematoxylin, cochineal, or iodeosin T.S. as an indicator. For this purpose a special experiment should be made, in which an accurately measured volume of 10 Cc. of the tenth-normal sulphuric acid, after adding 1 Cc. of hematoxylin T.S. (or a sufficient quantity of the indicator to be employed), should require 50 Cc. of the fiftieth-normal potassium hydroxide V.S. for complete neutralization at 25°C . (77°F .). If necessary, an adjustment should be made to render the correspondence perfect.

One Cubic Centimeter of Tenth-Normal Sulphuric Acid V.S. is the equivalent of:

	Gramme.
Sulphuric Acid, absolute, H_2SO_4	0.0048675
Potassium Hydroxide, KOH.....	0.005574
Aconitine, $\text{C}_{34}\text{H}_{47}\text{NO}_{11}$	0.06406
Atropine, $\text{C}_{17}\text{H}_{23}\text{NO}_3$	0.02870
Brucine, $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4$	0.03913
Calcium Hydroxide, $\text{Ca}(\text{OH})_2$	0.003678
Cephaeline, $\text{C}_{14}\text{H}_{19}\text{NO}_2$	0.02314
Cinchonidine, $\text{C}_{19}\text{H}_{22}\text{N}_2\text{O}$	0.02920
Cinchonine, $\text{C}_{19}\text{H}_{22}\text{N}_2\text{O}$	0.02920
Combined Alkaloids of Cinchona.....	0.03069
Combined Alkaloids of Ipecac.....	0.02384
Cocaine, $\text{C}_{17}\text{H}_{21}\text{NO}_4$	0.03009
Coniine, $\text{C}_8\text{H}_{17}\text{N}$	0.01262
Emetine, $\text{C}_{15}\text{H}_{21}\text{NO}_2$	0.02453
Hydrastine, $\text{C}_{21}\text{H}_{21}\text{NO}_8$	0.03803
Morphine, crystallized, $\text{C}_{17}\text{H}_{19}\text{NO}_3 + \text{H}_2\text{O}$	0.03009
Morphine, anhydrous, $\text{C}_{17}\text{H}_{19}\text{NO}_3$	0.02830
Physostigmine, $\text{C}_{15}\text{H}_{21}\text{N}_3\text{O}_2$	0.02732
Pilocarpine, $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_2$	0.02066
Quinine, $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2$	0.03218
Strychnine, $\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2$	0.03317

FIFTIETH-NORMAL SULPHURIC ACID VOLUMETRIC SOLUTION

 $\text{H}_2\text{SO}_4 = 97.35.$

0.9735 Gm. in 1000 Cc.

Dilute 20 Cc. of normal sulphuric acid V.S., or 200 Cc. of tenth-normal sulphuric acid V.S., with sufficient distilled water to measure at 25°C . (77°F .) 1000 Cc. This standard solution may be employed in the titration of alkaloids, with hematoxylin, cochineal, or iodeosin T.S. as an indicator.

TENTH-NORMAL OXALIC ACID VOLUMETRIC SOLUTION

 $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O} = 125.10.$

6.255 Gm. in 1000 Cc.

Dissolve 6.4 Gm. of pure oxalic acid in sufficient water to measure 1000 Cc.

Into a flask, accurately measure, from a burette, 10 Cc. of a *freshly standardized* tenth-normal potassium hydroxide V.S., dilute with about 20 Cc. of water, add 3 to 5 drops of phenolphthalein T.S., and heat to boiling. From a burette gradually add the oxalic acid solution (which is still too concentrated) until the red tint of the alkali solution fails to reappear after vigorous shaking and boiling. Note the number of Cc. of the oxalic acid solution consumed, and then dilute it so that equal volumes of this and of the tenth-normal potassium hydroxide V.S. neutralize each other at 25°C . (77°F .). It deteriorates on standing.

EXAMPLE.—Assuming that the 10 Cc. of the tenth-normal potassium hydroxide V.S. required exactly 9.5 Cc. of the oxalic acid solution, then each 9.5 Cc. of the latter must be diluted to 10 Cc., or the whole of the remaining acid solution in the same proportion at 25°C . (77°F .). Thus, if 950 Cc. of the oxalic acid solution should remain, 50 Cc. of water must be added.

After the liquid is thus diluted, a new trial should be made in the manner above

described, in which 50 Cc. of tenth-normal potassium hydroxide V.S. should require for neutralization exactly 50 Cc. of the oxalic acid solution at 25° C. (77° F.). If necessary, a new adjustment should then be made to render the correspondence perfect.

NOTE.—Tenth-normal oxalic acid V.S. is in every respect equivalent in neutralizing power to any other tenth-normal acid V.S. with either litmus or phenolphthalein T.S. as indicator. Its most important use is in standardizing tenth-normal potassium permanganate V.S.

One Cubic Centimeter of Tenth-Normal Oxalic Acid V.S. is the equivalent of:

	Gramme.
Oxalic Acid, crystallized, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$	0.006255
Ammonia Gas, NH_3	0.001693
Calcium Hydroxide, $\text{Ca}(\text{OH})_2$	0.003678
Lead Subacetate, $\text{Pb}_2\text{O}(\text{C}_2\text{H}_3\text{O}_2)_2$	0.0135935
Manganese Dioxide, precipitated, MnO_2	0.004318
Potassium Hydroxide, KOH	0.005574
Potassium Permanganate, KMnO_4	0.0031396
Sodium Hydroxide, NaOH	0.003976

NORMAL HYDROCHLORIC ACID VOLUMETRIC SOLUTION

$\text{HCl} = 36.18$.

36.18 Gm. in 1000 Cc.

Mix 130 Cc. of hydrochloric acid of specific gravity 1.158 with sufficient water to measure 1000 Cc.

Of this liquid (which is still too concentrated) carefully measure, from a burette, 10 Cc. into a flask or porcelain dish, and after diluting with about double its volume of water, add 2 drops of methyl-orange T.S., then gradually add, from a burette, a freshly standardized normal potassium hydroxide V.S., until the red tint of the solution changes, after vigorous shaking, to a permanent pale yellow. Note the number of Cc. of potassium hydroxide V.S. consumed, and then dilute the acid solution so that equal volumes of this and of the normal potassium hydroxide V.S. neutralize each other at 25° C. (77° F.).

EXAMPLE.—Assuming that 10 Cc. of the acid solution first prepared required exactly 11 Cc. of normal potassium hydroxide V.S., each 10 Cc. of the former must be diluted to 11 Cc., or the whole of the remaining acid solution in the same proportion at 25° C. (77° F.). Thus, if 950 Cc. should remain, 95 Cc. of water must be added.

After the liquid is thus diluted, a new trial should be made in the manner above described, in which 50 Cc. of the acid solution should require for neutralization exactly 50 Cc. of normal potassium hydroxide V.S. If necessary, a new adjustment should then be made to render the correspondence perfect at 25° C. (77° F.).

One Cubic Centimeter of Normal Hydrochloric Acid V.S. is the equivalent of:

	Gramme.
Hydrochloric Acid, absolute, HCl	0.03618

NOTE.—Normal hydrochloric acid is in every respect equivalent in neutralizing power to normal sulphuric acid (see p. 990), and may be employed, except in special cases, for the same purposes. However, preference is generally given to the normal sulphuric acid V.S.

HALF-NORMAL HYDROCHLORIC ACID VOLUMETRIC SOLUTION

$\text{HCl} = 36.18$.

18.09 Gm. in 1000 Cc.

Dilute 500 Cc. of normal hydrochloric acid V.S. with sufficient distilled water to measure exactly 1000 Cc. at 25° C. (77° F.).

One Cubic Centimeter of Half-Normal Hydrochloric Acid V.S. is the equivalent of:

	Gramme.
Hydrochloric Acid, absolute, HCl	0.01809
Benzaldehyde, $\text{C}_7\text{H}_6\text{O}$	0.0526
Cinnamic Aldehyde, $\text{C}_9\text{H}_8\text{O}$	0.0333
Citral, $\text{C}_{10}\text{H}_{16}\text{O}$	0.03802

NORMAL POTASSIUM HYDROXIDE VOLUMETRIC SOLUTION

KOH=55.74.

55.74 Gm. in 1000 Cc.

Dissolve 75 Gm. of potassium hydroxide [*Potassii Hydroxidum*, U. S. P.] in sufficient water to measure about 1050 Cc., and fill a burette with a portion of this liquid.

Into a flask of the capacity of about 300 Cc., introduce 9.339 Gm. of potassium bitartrate, which has been purified and dried, and 160 Cc. of distilled water. Boil the liquid until solution has taken place, add from 3 to 5 drops of phenolphthalein T.S., followed by the cautious addition, from a burette, of the potassium hydroxide solution, frequently agitating the flask, boiling, and, toward the end of the operation, reducing the flow to drops until the red color produced by its influx no longer disappears on shaking, but is not deeper than pale pink. Note the number of Cc. of the potassium hydroxide solution consumed, and then dilute the remainder of the solution so that exactly 50 Cc. of the diluted liquid at 25° C. (77° F.) shall be required to neutralize the 9.339 Gm. of potassium bitartrate used.

EXAMPLE.—Assuming that 40 Cc. of the stronger solution of potassium hydroxide first prepared had been consumed in the trial, then each 40 Cc. must be diluted to 50 Cc., or the whole of the remaining solution in the same proportion at 25° C. (77° F.). Thus, if 1000 Cc. should be still remaining, this must be diluted with water to 1250 Cc.

After the liquid is thus diluted, a new trial should be made in the manner above described, in which 50 Cc. of the diluted solution should exactly neutralize 9.339 Gm. of potassium bitartrate at 25° C. (77° F.). If necessary, a new adjustment should then be made to render the correspondence perfect.

NOTE.—Solutions of caustic alkalis absorb carbon dioxide from the atmosphere, and thereby change their titer when used with litmus T.S., or phenolphthalein T.S., as indicator (methyl-orange T.S. is not affected by the presence of carbonic acid). Hence the volumetric solutions should be preserved in bottles provided with well-fitting rubber stoppers, or, better still, these should be provided with tubes filled with soda-lime (a mixture of caustic soda and lime); the tubes pass through a perforation in the rubber stoppers, and thus absorb the carbon dioxide and prevent its access to the solution. If the solution is kept in a burette for any length of time, the same provision with a soda-lime tube should be observed.

In place of potassium hydroxide V.S., sodium hydroxide V.S. (see p. 995) may be used, in the same manner and in the same quantity. Potassium hydroxide V.S., however, is preferable, since it foams less and attacks glass more slowly.

One Cubic Centimeter of Normal Potassium Hydroxide V.S. is the equivalent of:

	Gramme.
Potassium Hydroxide, KOH.....	0.05574
Acetic Acid, absolute, $\text{HC}_2\text{H}_3\text{O}_2$	0.05958
Ammonia Gas, NH_3	0.01693
Ammonium Chloride, NH_4Cl	0.05311
Boric Acid, H_3BO_3	0.06154
Citric Acid, crystallized, $\text{H}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$	0.06950
Hydriodic Acid, absolute, HI.....	0.12690
Hydrobromic Acid, absolute, HBr.....	0.08036
Hydrochloric Acid, absolute, HCl.....	0.03618
Hypophosphorous Acid, $\text{H}_3\text{P}_2\text{O}_5$	0.06553
Lactic Acid, absolute, $\text{HC}_3\text{H}_5\text{O}_3$	0.08937
Nitric Acid, absolute, HNO_3	0.06257
Oxalic Acid, crystallized, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$	0.06255
Phosphoric Acid, H_3PO_4 (to form K_2HPO_4 ; with phenolphthalein)....	0.048645
Potassium Dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$	0.14614
Sodium Hydroxide, NaOH.....	0.03976
Sulphuric Acid, absolute, H_2SO_4	0.048675
Tartaric Acid, crystallized, $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	0.07446
Trichloroacetic Acid, CCl_3COOH	0.16212

TENTH-NORMAL POTASSIUM HYDROXIDE VOLUMETRIC SOLUTION

KOH=55.74.

5.574 Gm. in 1000 Cc.

Dilute 100 Cc. of freshly standardized normal potassium hydroxide V.S. with sufficient distilled water to measure, at 25° C. (77° F.), exactly 1000 Cc.

This solution may also be prepared and standardized directly with potassium bitartrate, as directed under normal potassium hydroxide V.S., employing for this purpose 0.9339 Gm. of the former and a solution of about 7.5 Gm. of potassium hydroxide [*Potassii Hydroxidum*, U. S. P.] in 1000 Cc. of distilled water. Fifty Cc. of the prepared tenth-normal solution at 25° C. (77° F.) should exactly neutralize 0.9339 Gm. of potassium bitartrate.

NOTE.—The same precautions should be taken for protecting this solution from carbon dioxide of the air as are directed under normal potassium hydroxide V.S.

One Cubic Centimeter of Tenth-Normal Potassium Hydroxide V.S. is the equivalent of:

	Gramme.
Potassium Hydroxide, KOH.....	0.005574
Sulphuric Acid, absolute, H ₂ SO ₄	0.0048675

FIFTIETH-NORMAL POTASSIUM HYDROXIDE VOLUMETRIC SOLUTION

KOH=55.74. 1.1148 Gm. in 1000 Cc.

Dilute 20 Cc. of a freshly standardized normal or 200 Cc. of tenth-normal potassium hydroxide V.S. with sufficient distilled water to measure at 25° C. (77° F.) exactly 1000 Cc. This standard solution is employed in conjunction with the tenth-normal sulphuric acid V.S. in the titration of alkaloids, with hematoxylin, cochineal, or iodeosin T.S. as indicators.

NOTE.—The same precautions should be taken for protecting this solution from carbon dioxide of the air as are directed under normal potassium hydroxide V.S. It should be renewed at frequent intervals.

HUNDREDTH-NORMAL POTASSIUM HYDROXIDE VOLUMETRIC SOLUTION

KOH=55.74. 0.5574 Gm. in 1000 Cc.

Dilute 10 Cc. of normal or 100 Cc. of tenth-normal potassium hydroxide V.S. with sufficient water to measure, at 25° C. (77° F.), exactly 1000 Cc. The solution must be frequently renewed.

One Cubic Centimeter of Hundredth-Normal Potassium Hydroxide V.S. is the equivalent of:

	Gramme.
Potassium Hydroxide, KOH.....	0.0005574
Sulphuric Acid, absolute, H ₂ SO ₄	0.00048675

HALF-NORMAL ALCOHOLIC POTASSIUM HYDROXIDE VOLUMETRIC SOLUTION

KOH=55.74. 27.87 Gm. in 1000 Cc.

Dissolve about 40 Gm. of potassium hydroxide [*Potassii Hydroxidum*, U. S. P.], which has been broken into small pieces, in about 20 Cc. of water, and add sufficient alcohol [*Alcohol*, U. S. P.] to measure 1000 Cc. After setting aside in a well-stoppered bottle for one day, the clear supernatant solution should be quickly decanted into a bottle provided with a well-fitted rubber stopper.

Into a flask of the capacity of about 300 Cc. introduce 1.8678 Gm. of potassium bitartrate, which has been purified and dried, and about 100 Cc. of distilled water. Heat the solution to the boiling-point, add 5 drops of phenolphthalein T.S., followed by the cautious addition, from a burette, of the potassium hydroxide solution, frequently agitating the flask, boiling, and, toward the end of the operation, reducing the flow to drops until the red color produced by its influx no longer disappears on shaking, but is not deeper than pale pink. Note the number of Cc. of the alcoholic potassium hydroxide solution consumed, and then dilute the remainder of the solution with alcohol so that exactly 20 Cc. of the diluted liquid shall be required to neutralize the 1.8678 Gm. of potassium bitartrate used.

EXAMPLE.—Assuming that 12.5 Cc. of the stronger alcoholic solution of potassium hydroxide first prepared has been consumed in the trial, then each 12.5 Cc. must be diluted with alcohol to 20 Cc., or the whole of the remaining solution in the same proportion at 25° C. (77° F.). Thus, if 980 Cc. should remain, this must be diluted with alcohol to measure 1568 Cc.

Half-Normal Alcoholic Potassium Hydroxide V.S. may also be prepared as fol-

lows: Of the above-described concentrated alcoholic potassium hydroxide solution, carefully measure, from a burette, 20 Cc. into a flask, and, after diluting with about 50 Cc. of water, add about 5 drops of phenolphthalein T.S., heat to a boiling temperature, and add, from a burette, half-normal hydrochloric acid V.S., frequently agitating the flask, and toward the end of the operation, reducing the flow to drops until the red color is just discharged. Note the number of Cc. of the half-normal hydrochloric acid V.S. consumed, and then dilute the remainder of the solution with alcohol so that equal volumes of this and of the half-normal hydrochloric acid V.S. neutralize each other at 25° C. (77° F.).

Should half-normal hydrochloric acid V.S. not be available, standardization can be carried out in the same manner with *normal* hydrochloric acid V.S., two volumes of the alcoholic potassium hydroxide being made to correspond with one volume of the standard acid.

NOTE.—This solution should be kept in bottles provided with a well-fitted rubber stopper and protected from the light. Owing to the readiness with which this standard solution loses its titer, blank tests should be performed whenever it is employed in titrations.

One Cubic Centimeter of Half-Normal Alcoholic Potassium Hydroxide V.S. is the equivalent of:

	Gramme.
Potassium Hydroxide, KOH.....	0.02787
Borneol, $C_{10}H_{18}O$	0.07649
Bornyl Acetate, $C_{10}H_{17}O.C_2H_3O$	0.09734
Menthol, $C_{10}H_{20}O$	0.07749
Menthyl Acetate, $C_{10}H_{19}O.C_2H_3O$	0.09834
Santalol, $C_{15}H_{26}O$	0.11026

NORMAL SODIUM HYDROXIDE VOLUMETRIC SOLUTION

NaOH = 39.76. 39.76 Gm. in 1000 Cc.

Dissolve 54 Gm. of sodium hydroxide [*Sodii Hydroxidum*, U. S. P.] in sufficient water to measure about 1050 Cc., and fill a burette with a portion of this liquid.

For the standardization of this approximate solution of sodium hydroxide, proceed as directed under normal potassium hydroxide V.S. (see p. 993); 50 Cc. of the volumetric solution, at 25° C. (77° F.), must exactly neutralize 9.339 Gm. of pure potassium bitartrate.

NOTE.—The same precautions should be taken for protecting this solution from the carbon dioxide of the air as are prescribed for normal potassium hydroxide V.S. (see p. 993).

This solution may be employed in place of the normal potassium hydroxide V.S. (see p. 993), volume for volume.

DOUBLE-NORMAL SODIUM HYDROXIDE VOLUMETRIC SOLUTION

NaOH = 39.76. 79.52 Gm. in 1000 Cc.

Dissolve 70 Gm. of sodium hydroxide [*Sodii Hydroxidum*, U. S. P.] in sufficient water to measure about 1000 Cc.

For the standardization of this approximate solution of sodium hydroxide proceed as directed under normal potassium hydroxide V.S. (see p. 993); 25 Cc. of the volumetric solution, at 25° C. (77° F.), should exactly neutralize 9.339 Gm. of pure potassium bitartrate.

NOTE.—The same precautions should be taken for protecting this solution from carbon dioxide of the air as are prescribed for normal potassium hydroxide V.S. (see p. 993).

In preparing these volumetric solutions the dry chemicals are weighed on an appropriate scale, the finished standardized solution brought to the exact volume in an accurately measured flask (see Fig. 19, of 1000-Cc. flask and 100-Cc. flask on p. 45). The other apparatus required in volumetric analysis are pipettes (see Fig. 20, p. 45), burettes, which have just been described, and a number of chemical flasks of appropriate size.

OFFICIAL CHEMICALS ASSAYED WITH NORMAL POTASSIUM HYDROXIDE V. S.

Hydrochloric Acid.—Introduce into a stoppered weighing-bottle 3 Cc. of Hydrochloric Acid and weigh accurately. Dilute the Acid with 50 Cc. of distilled water and titrate with normal potassium hydroxide V.S., using methyl-orange T.S. as indicator. Multiply the number of Cc. of the normal potassium hydroxide V.S. consumed by 3.618, and divide this product by the weight of the Acid taken; the quotient represents the percentage of absolute Hydrochloric Acid in the latter.

It will be seen that the mathematic equation used in calculating strength is $\frac{\text{Cc. nKOH used} \times 3.618}{\text{weight of acid}} = \text{per cent. absolute acid.}$

This equation is used for the three acids which follow, the only difference being that the factor 3.618 is changed in each case for the one appropriate to the acid in question. Referring to the table of values under normal Potassium Hydroxide V.S. on p. 993, it will be seen that 1 Cc. of the volumetric solution equals 0.03618 Gm. absolute hydrochloric acid. This, multiplied by 100 (100 per cent.), equals 3.618—the factor given. In similar manner are the factors of the three other acids derived.

Nitric Acid.—Like hydrochloric acid assay, except factor is 6.257.

Sulphuric Acid.—Like hydrochloric acid assay, except factor is 4.8675.

Glacial Acetic Acid.—Like hydrochloric acid assay, except factor is 5.958, and that phenolphthalein is the indicator employed.

Diluted Hydrochloric Acid.—If to 3.62 Gm. of Diluted Hydrochloric Acid there be added about 20 Cc. of water, it should require 10 Cc. of normal potassium hydroxide V.S. for neutralization (each Cc. corresponding to 1 per cent. of absolute Hydrochloric Acid), methyl-orange T.S. being used as indicator.

In the case of this assay and the three assays which follow it the quantity of the diluted acid equivalent to 100 times the amount of absolute acid neutralized by 1 Cc. normal potassium hydroxide V.S. is weighed out, and this amount should take exactly 10 Cc. nKOH to neutralize it, as is shown in the following calculation:

If 1 Cc. nKOH = 0.03618 Gm. absolute HCl (p. 993),
Then 10 Cc. nKOH = 10×0.03618 Gm. absolute HCl
or 0.3618 Gm. absolute HCl.

Then 3.62 Gm. diluted acid contains 0.3618 Gm. absolute acid, and is, therefore, 10 per cent.

The diluted acetic acid calculation is different and is given below. The four other acids tested like diluted hydrochloric acid are:

ACID.	AMOUNT USED.	DILUTED WITH.	REQUIRES Cc. nKOH.	INDICATOR.	STRENGTH OF ACID IS—
<i>Diluted nitric acid.</i>	6.26 Gm.	Nothing.	10 Cc.	Methyl orange.	10 per cent.
<i>Diluted sulphuric acid.</i>	4.86 Gm.	Nothing.	10 Cc.	Methyl orange.	10 per cent.
<i>Diluted phosphoric acid.</i>	4.87 Gm.	Saturated solution of potassium chloride (5 Cc.).	10 Cc.	Phenolphthalein.	10 per cent.
<i>Diluted acetic acid.</i>	23.8 Gm.	Nothing.	24 Cc.	Phenolphthalein.	6 per cent.
<i>Tartaric acid.</i>	3.728 Gm.	Presumably water.	49.8 Cc.	Phenolphthalein.	99.6 per cent.

The calculation for acetic acid is as follows:

1 Cc. nKOH = 0.05958 Gm. absolute acetic acid (p. 993).
 24 Cc. nKOH = 24×0.05958 Gm. absolute acetic acid.
 1.42992 Gm. absolute acetic acid.

Hence, 23.8 Gm. diluted acetic acid contains 1.43992 Gm. absolute acid, and the percentage of absolute acid in the diluted acid is, therefore,

$$\frac{1.43992}{23.80000} \times 100 \text{ per cent.} = 6 \text{ per cent.}$$

As the amount of tartaric acid used is 50 times the quantity which 1 Cc. nKOH will neutralize, each Cc. nKOH in such case will represent 2 per cent. tartaric acid.

Phosphoric Acid.—If 10 Gm. of Phosphoric Acid be diluted to measure 100 Cc., then 9.73 Cc. of this solution, when diluted with 10 Cc. of a cold saturated solution of sodium chloride, should require 17 Cc. of normal potassium hydroxide V.S. for neutralization (each Cc. corresponding to 5 per cent. of absolute Phosphoric Acid), phenolphthalein T.S. being used as indicator.

In this and the five following assays, instead of weighing the exact amount of acid as in the case of diluted hydrochloric acid, a 10 per cent. (in one case, 5 per cent.) dilution is prepared, and an aliquot part of this dilution, representing some multiple of the normal potassium hydroxide V.S. value of the tested acid, is taken. This is best shown by calculating the phosphoric acid figures just given.

9.73 Cc. of a 10 per cent. dilution of U. S. P. phosphoric acid contains 0.973 Gm. U. S. P. phosphoric acid.

1 Cc. nKOH = 0.048645 Gm. absolute H_3PO_4 (p. 993).
 17 Cc. nKOH = 17×0.048645 Gm. absolute H_3PO_4 .
 = 0.826965 Gm. absolute H_3PO_4 .

Then the percentage of absolute H_3PO_4 in 0.973 Gm. U. S. P. acid is—

$$\frac{0.826965}{0.973000} \times 100 \text{ per cent.} = 85 \text{ per cent.}$$

The five other acids assayed like phosphoric acid are:

ACID.	AMOUNT AND STRENGTH OF DILUTION.	REQUIRES Cc. nKOH.	INDICATOR.	STRENGTH OF ACID IS—
<i>Hypophosphorous acid.</i>	65.5 Cc. of 10 per cent. dilution.	30 Cc.	Methyl orange.	30 per cent.
<i>Diluted hypophosphorous acid.</i>	65.5 Cc. of 10 per cent. dilution.	10 Cc.	Methyl orange.	10 per cent.
<i>Acetic acid.</i>	59.6 Cc. of 10 per cent. dilution.	36 Cc.	Phenolphthalein.	36 per cent.
<i>Lactic acid.</i>	44.7 Cc. of 10 per cent. dilution.	37.5 Cc.	Phenolphthalein.	75 per cent.
<i>Citric acid.</i>	34.75 Cc. of 5 per cent. dilution.	24.87 Cc.	Phenolphthalein.	99.48 per cent.

In the above assays the amount of acid used in assay is, in the case of phosphoric acid, 20 times the nKOH value; in the case of citric acid, is 25 times the nKOH value; in the case of lactic acid, is 50 times the nKOH value, and in the case of the rest, is 100 times the nKOH value.

The assays of the following acids with normal potassium hydroxide V.S. are too intricate for tabulation, hence are given in full:

Aromatic Sulphuric Acid.—If 10 Gm. of Aromatic Sulphuric Acid be mixed in a small flask with 30 Cc. of water and be boiled for four hours, cooled and diluted with water to measure 100 Cc., then 48.68 Cc. of this solution, to which exactly 25 Cc. of normal potassium hydroxide V.S. have been added, should require not more than 5 Cc. of normal sulphuric acid V.S. for complete neutralization (each Cc. of the normal potassium hydroxide V.S. consumed corresponding to 1 per cent. of absolute Sulphuric Acid), methyl-orange T.S. being used as indicator.

Lemon Juice.—At least 10 Cc. of normal potassium hydroxide V.S. should be required to neutralize 10 Cc. of Lemon juice, phenolphthalein T.S. being used as indicator.

OFFICIAL CHEMICALS ASSAYED WITH NORMAL SODIUM HYDROXIDE V. S.

Boric Acid.—One Gm. of Boric Acid, when dissolved in 50 Cc. of distilled water, after the addition of 50 Cc. of glycerin, should require not less than 16.2 Cc. of normal sodium hydroxide V.S. for neutralization (corresponding to at least 99.8 per cent. of Boric Acid), phenolphthalein T.S. being used as indicator.

Trichloroacetic Acid.—One Gm. when diluted with 50 Cc. of water should not require less than 6.1 Cc. of normal sodium hydroxide V.S. for neutralization, phenolphthalein T.S. being used as indicator.

Solution of Formaldehyde.—Transfer 3 Cc. of Solution of Formaldehyde to a well-stoppered Erlenmeyer flask, and weigh accurately. Add 50 Cc. of normal sodium hydroxide V.S., and follow this immediately, but slowly, through a small funnel, with 50 Cc. of solution of hydrogen dioxide, to which a drop of litmus T.S. has been added, and which has been neutralized with normal sodium hydroxide V.S. After the reaction has ceased and the foaming has subsided, rinse the funnel and sides of the vessel with distilled water, and, after allowing it to stand ten minutes, titrate back with normal sulphuric acid V.S., using litmus T.S. as indicator. Subtract the number of Cc. of normal sulphuric acid V.S. consumed from 50 (the number of Cc. of normal sodium hydroxide V.S. employed), multiply the remainder by 2.979, and divide the product by the weight of the Solution taken; the quotient represents the percentage, by weight, of absolute Formaldehyde in the liquid.

This assay depends on the oxidation of formaldehyde by hydrogen dioxide to formic acid, which is then titrated with normal soda.

OFFICIAL CHEMICALS ASSAYED WITH NORMAL SULPHURIC ACID V. S.

Potassium Hydroxide.—Introduce about 1 Gm. of Potassium Hydroxide into a stoppered weighing-bottle and weigh accurately. Dissolve this in about 50 Cc. of water and titrate the solution with normal sulphuric acid V.S., using methyl-orange T.S. as indicator. Multiply the number of Cc. of the normal sulphuric acid V.S. consumed by 5.574, and divide this product by the weight of the Potassium Hydroxide taken; the quotient, which must be not less than 85, represents the percentage of Potassium Hydroxide present.

It will be seen that the equation used in calculating the percentage of absolute KOH is—

$$\frac{\text{Cc. nH}_2\text{SO}_4 \text{ used} \times 5.574}{\text{weight of KOH used}} = \text{per cent. absolute KOH.}$$

The factor 5.574 is 100 times the quantity of KOH which 1 Cc. nH₂SO₄ will neutralize (see p. 990), hence the reasoning used in calculation is the same as that given under the assay of hydrochloric acid (p. 996).

Sodium Hydroxide.—Like potassium hydroxide assay except factor is 3.976.

Ammonia Water.—

Stronger Ammonia Water.— $\left\{ \begin{array}{l} \text{Like potassium hydroxide assay except factor is} \\ 1.693, \text{ and 3 Gm. of the solution is weighed ac-} \\ \text{curately.} \end{array} \right.$

Solution of Potassium Hydroxide.—To neutralize 28 (27.87) Gm. of Solution of Potassium Hydroxide should require about 25 Cc. of normal sulphuric acid V.S., methyl-orange T.S. being used as indicator (each Cc. of normal sulphuric acid V.S. indicating 0.2 per cent. of absolute Potassium Hydroxide).

This assay is based on the same principles as the assay of diluted hydrochloric acid (p. 996); the amount of solution of potassa taken being 500 times the amount of absolute KOH neutralized by 1 Cc. nH_2SO_4 . (See p. 990.)

Solution of Sodium Hydroxide.—Like assay of solution of potassium hydroxide, except that 20 Gm. of solution (500 times the amount of absolute NaOH neutralized by 1 Cc. nH_2SO_4) is taken.

Potassium Carbonate.—One Gm. of Potassium Carbonate, weighed after having been thoroughly dried at 130°C . (266°F .), and dissolved in about 50 Cc. of water, should require not less than 14.3 (14.28) Cc. of normal sulphuric acid V.S. for neutralization, methyl-orange T.S. being used as indicator.

Sodium Bicarbonate.—Two Gm. of Sodium Bicarbonate should require for complete neutralization not less than 23.7 (23.74) Cc. of normal sulphuric acid V.S., methyl-orange T.S. being used as indicator.

Lithium Carbonate.—If 0.5 Gm. of Lithium Carbonate be dissolved in 20 Cc. of normal sulphuric acid V.S., the resulting solution should require not more than 6.6 Cc. of normal potassium hydroxide V.S. for complete neutralization, methyl-orange T.S. being used as indicator.

In this case the salt is dissolved in definite quantity of nH_2SO_4 , and the excess of acid estimated by titration with nKOH . Since 6 Cc. nKOH equals 6 Cc. nH_2SO_4 , it is evident that the carbonate takes 14 Cc. (20—6) nH_2SO_4 to neutralize it.

The following official salts are assayed like lithium carbonate, the difference in quantity of salt and of volumetric solution used being given in tabulated form:

SALT.	AMOUNT USED.	Cc. nH_2SO_4 USED FOR SOLUTION.	Cc. nKOH NEEDED TO NEUTRALIZE EXCESS OF ACID.	INDICATOR.
<i>Ammonium carbonate.</i>	2 Gm.	$\left\{ \begin{array}{l} 50 \text{ Cc. } \text{nH}_2\text{SO}_4.^1 \\ 50 \text{ Cc. water.} \end{array} \right.$	12.7 Cc.	Litmus T.S.
<i>Magnesium carbonate</i> (recently ignited).	0.4 Gm.	25 Cc.	5.8 Cc.	Methyl orange.
<i>Magnesium oxide</i> (recently ignited).	0.4 Gm.	25 Cc.	5.8 Cc.	Methyl orange.

OFFICIAL CHEMICALS ASSAYED WITH HALF-NORMAL SULPHURIC ACID V. S.

Potassium Acetate.—If 1 Gm. of dry Potassium Acetate be thoroughly carbonized at a temperature not exceeding red heat, and the residue extracted with boiling distilled water until the washings cease to react with methyl-orange T.S., the mixed filtrate and washings should require for complete neutralization not less than 20.1 Cc. of half-normal sulphuric acid V.S., methyl-orange T.S. being used as indicator.

The following official chemicals are assayed like potassium acetate:

	AMOUNT SALT USED.	Cc. $\frac{1}{2} \text{ nH}_2\text{SO}_4$ REQUIRED.	INDICATOR.
<i>Potassium bitartrate.</i>	1 Gm.	10.6 Cc.	Methyl orange.
<i>Solution of potassium citrate.</i>	10.14 Gm.	16.0 Cc.	Methyl orange.
<i>Sodium acetate.</i>	1 Gm.	14.7 Cc.	Methyl orange.
<i>Sodium citrate.</i>	1 Gm.	16.4 Cc.	Methyl orange.
<i>Sodium salicylate.</i>	1 Gm.	12.5 Cc.	Methyl orange.

¹ The acid solution of ammonium carbonate must be boiled until all the carbon dioxide is expelled.

Potassium Bicarbonate.—To neutralize 1 Gm. of Potassium Bicarbonate not less than 19.9 Cc. of half-normal sulphuric acid V.S. should be required, methyl-orange T.S. being used as indicator.

Monohydrated Sodium Carbonate.—If 1 Gm. of Monohydrated Sodium Carbonate or 0.855 Gm. of the anhydrous salt, be dissolved in 10 Cc. of distilled water, it should require not less than 32.3 Cc. of half-normal sulphuric acid V.S. for neutralization, 3 drops of methyl-orange T.S. being used as indicator.

Spirit of Ammonia.—Assayed like ammonia water (p. 998), except that 2 Gm. spirit is weighed, $\frac{1}{2}$ NH_4SO_4 used, and the factor is 0.008465×100 .

OFFICIAL CHEMICAL ASSAYED WITH TENTH-NORMAL SULPHURIC ACID V.S.

Solution of Lime.—Fifty Cc. should require, for complete neutralization, not less than 19 Cc. of tenth-normal sulphuric acid V.S. (corresponding to about 0.14 per cent. of calcium hydroxide), phenolphthalein T.S. being used as indicator.

OFFICIAL CHEMICALS ASSAYED WITH HALF-NORMAL HYDROCHLORIC ACID V.S.

The following are assayed like potassium acetate (p. 999):

Potassium Citrate.—One Gm. after ignition takes not less than 18.4 Cc. $\frac{1}{2}$ NHCl .

Potassium and Sodium Tartrate.—One Gm. after ignition takes not less than 14.1 Cc. $\frac{1}{2}$ NHCl .

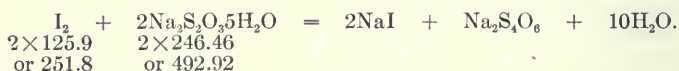
Sodium Benzoate.—One Gm. after ignition takes not less than 13.85 Cc. $\frac{1}{2}$ NHCl .

Zinc Oxide.—If 1 Gm. of freshly ignited Zinc Oxide be digested with 30 Cc. of normal hydrochloric acid V.S. until solution is complete, and 2 drops of methyl-orange T.S. be added, not more than 5.4 Cc. of normal potassium hydroxide V.S. should be required for neutralization (each Cc. of normal hydrochloric acid consumed corresponding to about 4 per cent. of zinc oxide). The normal potassium hydroxide V.S. should be added slowly with constant stirring, waiting until the precipitated zinc hydroxide has redissolved before adding further portions of the reagent.

IODOMETRY

By this term we mean those volumetric assays in which we use volumetric solution of iodine or volumetric solution of sodium thiosulphate, or both.

As already explained (p. 396), iodine in solution is decolorized by use of sodium thiosulphate, and this decoloration, like all chemical actions, takes place between definite proportions of the two chemicals, the equation being:



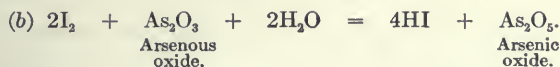
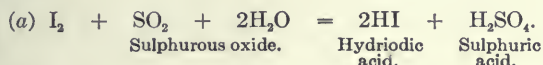
We see from this equation that 251.8 Gm. iodine take 492.92 Gm. crystallized sodium thiosulphate to complete the reaction—to completely decolorize it. Therefore, if we prepare a decinormal iodine solution (12.59 Gm. to the liter), we can calculate that this 12.59 Gm.

iodine will take $\frac{12.59}{251.80} \times 492.92 = 24.646$ Gm. sodium thiosulphate,

which amount is exactly the quantity needed to make 1 liter of decinormal sodium thiosulphate solution. Hence equal volumes of the two decinormal solutions exactly react with each other.

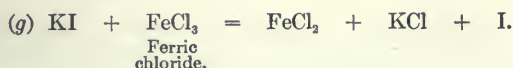
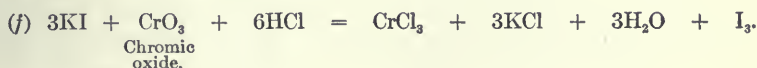
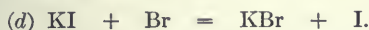
In titrating iodine solution with thiosulphate solution we either add the thiosulphate solution until the brown-yellow color of the iodine is discharged, or, better still, we use as indicator starch mucilage, which we have already learned strikes a deep-blue color with iodine (p. 396). The moment all the iodine in a given solution is taken up by the thiosulphate, the blue color is discharged.

Were the sole use of iodine solution and thiosulphate solution the estimation of the strength of these two chemicals, the value of iodometry would be limited. Far more extended, however, is its application, because of the fact that iodine, when treated with reducing agents, is converted into hydriodic acid or iodides, as shown in the following equations:



and thus loses its ability to color starch mucilage blue. Hence if we treat a known excessive quantity of iodine solution with any of the reducing agents just mentioned, by titrating the excess of iodine remaining after the reaction with thiosulphate solution, we can calculate how much iodine was used by the chemical and thus estimate the amount of chemical itself.

On the other hand, potassium iodide when treated with oxidizing agents liberates a proportional quantity of its iodine, as is shown in the following equations:



and by titrating this liberated iodine with thiosulphate solution the amount of the oxidizing agent can be deduced.

The pharmacopœial preparation of decinormal iodine solution and of decinormal thiosulphate solution are given below.

TENTH-NORMAL IODINE VOLUMETRIC SOLUTION

 $I = 125.9.$

12.59 Gm. in 1000 Cc.

Tenth-normal iodine V.S. may be prepared according to either of the following methods:

I. Dissolve 12.59 Gm. of pure iodine (see below) in a solution of 18 Gm. of potassium iodide in 300 Cc. of water. Then add sufficient water to make the solution measure, at 25° C. (77° F.), exactly 1000 Cc. Unless freshly prepared, its strength should always be determined anew at the time it is used. Transfer the solution to glass-stoppered vials.

Preparation of Pure Iodine.—Heat powdered iodine [*Iodum*, U. S. P.] in a porcelain dish placed over a bath of boiling water for twenty minutes, and stir it constantly with a glass rod, so that adhering moisture, cyanogen iodide, and most of the iodine bromide and iodine chloride, if present, may be vaporized. Then transfer the iodine to a porcelain or other non-metallic mortar, and triturate it with about 5 per cent. of its weight of dry potassium iodide, so as to decompose any remaining iodine bromide and iodine chloride. Then return the mass to the dish, cover it with a glass funnel, and heat the dish carefully on a sand-bath. Detach the sublimed,

pure iodine, and, after pulverizing and drying for twenty-four hours over calcium chloride, keep it in well-stoppered bottles, in a cool place.

II. Tenth-normal iodine V.S. may also be prepared as follows:

Dissolve about 14 Gm. of iodine [*Iodum*, U. S. P.] in a solution of 18 Gm. of potassium iodide [*Potassii Iodidum*, U. S. P.] in about 300 Cc. of water, diluting finally to 1000 Cc. Of this solution (which is too concentrated), carefully measure from a burette 10 Cc. in a flask, then add gradually and cautiously from a burette tenth-normal sodium thiosulphate V.S. (shaking constantly) until the color of the solution is discharged. Note the number of Cc. of the sodium thiosulphate V.S. consumed, and then dilute the iodine solution so that any known volume of the latter will require for decolorization exactly the same volume of the tenth-normal sodium thiosulphate V.S.

EXAMPLE.—Assuming that 10 Cc. of the iodine solution required 10.8 Cc. of the tenth-normal sodium thiosulphate V.S. for decolorization, then each 10 Cc. of the former must be diluted to 10.8 Cc., or each 100 Cc. of the iodine solution to 108 Cc. at 25° C. (77° F.). After the solution is thus diluted, a new trial should be made in the manner above described, in which 50 Cc. of the tenth-normal iodine V.S. should require exactly 50 Cc. of the tenth-normal sodium thiosulphate V.S. for complete decolorization. If necessary, a new adjustment should be made to render the correspondence perfect.

One Cubic Centimeter of Tenth-Normal Iodine V.S. is the equivalent of:

	Gramme.
Iodine, I.....	0.01259
Arsenic, As.....	0.00372
Arsenic Trioxide (Arsenous acid), As ₂ O ₃	0.004911
Iron, Fe.....	0.002775
Potassium Sulphite, crystallized, K ₂ SO ₃ + 2H ₂ O.....	0.009648
Sodium Bisulphite, NaHSO ₃	0.005168
Sodium Thiosulphate (Hyposulphite), crystals, Na ₂ S ₂ O ₃ + 5H ₂ O.....	0.024646
Sodium Sulphite, crystallized, Na ₂ SO ₃ + 7H ₂ O.....	0.012520
Sulphur Dioxide, SO ₂	0.003180
Antimony and Potassium Tartrate, crystallized, 2K(SbO)C ₄ H ₄ O ₆ + H ₂ O.....	0.016495

TENTH-NORMAL SODIUM THIOSULPHATE VOLUMETRIC SOLUTION



24.646 Gm. in 1000 Cc.

Dissolve 30 Gm. of sodium thiosulphate in sufficient distilled water to measure 1000 Cc. This trial solution, which is too concentrated, is standardized as follows:

To a solution of about 1 Gm. of potassium iodide [*Potassii Iodidum*, U. S. P.] in 10 Cc. of diluted sulphuric acid contained in a flask of about 500-Cc. capacity, add slowly, from a burette, 20 Cc. of tenth-normal potassium dichromate V.S., shaking after each addition. Place a watch glass on the mouth of the flask and allow it to stand for five minutes, then dilute the solution with about 250 Cc. of distilled water, add some starch T.S., and then, from a burette, the trial solution of sodium thiosulphate, in small portions at a time, shaking after each addition, and, toward the end of the operation, reducing the flow to drops, until the blue color of the mixture changes to a light green; note the number of Cc. of the trial sodium thiosulphate solution consumed. Then dilute the sodium thiosulphate solution so that equal volumes of it and the tenth-normal potassium dichromate V.S. will exactly correspond to each other under the above conditions at 25° C. (77° F.).

EXAMPLE.—Assuming that 16 Cc. of the trial sodium thiosulphate solution were required to decolorize the liberated iodine of the mixture, then each 16 Cc. of this solution must be diluted to 20 Cc., so that it will correspond in volume to the tenth-normal potassium dichromate V.S. added, or the whole of the remaining solution in the same proportion at 25° C. (77° F.). Thus, if 984 Cc. of the sodium thiosulphate solution are remaining, this should be diluted to measure 1230 Cc. After the sodium thiosulphate solution is thus diluted, a new trial should be made in the manner above described, in which exactly 20 Cc. of this solution should be required to decolorize the iodine liberated by 20 Cc. of the tenth-normal dichromate V.S. If necessary, a new adjustment should be made to render the correspondence perfect.

Keep the solution in glass-stoppered bottles, carefully protected from dust.

NOTE.—When this solution is to be used, fill a burette with it, place the liquid to be tested either for the free iodine it already contains, or for that which it liberates from an excess of potassium iodide added to it, in a flask, and gradually add small

portions of the solution from the burette, shaking after each addition, and, toward the end of the operation, reducing the flow to drops, until the color is discharged.

One Cubic Centimeter of Tenth-Normal Sodium Thiosulphate V.S. is the equivalent of:

	Gramme.
Sodium Thiosulphate (Hyposulphite), $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$	0.024646
Bromine, Br.....	0.007936
Chlorine, Cl.....	0.003518
Chromium Trioxide, CrO_3	0.003311
Iodine, I.....	0.01259
Iron, Fe, in ferric salts.....	0.00555
Potassium Bromate, KBrO_3	0.002764

A comparison of the several iodine equations given above with the amount of the characteristic chemical represented by 1 Cc. $\frac{1}{10}$ n iodine solution or by 1 Cc. $\frac{1}{10}$ n thio solution may be timely. Already we have calculated (p. 1000) that 1 Cc. $\frac{1}{10}$ n thio = 1 Cc. $\frac{1}{10}$ n iodine. Just above we learned that 1 Cc. $\frac{1}{10}$ n thio contains 0.01259 Gm.; that is, the same number of grammes of iodine as its atomic weight divided by 10,000. Likewise 1 Cc. of $\frac{1}{10}$ n thio contains 0.024646 Gm. of sodium thiosulphate; that is, the same number of grammes of thio as its molecular weight divided by 10,000.

From the equations given on p. 1001 we find:

Equation "a" 1 atom iodine equals $\frac{1}{2}$ molecule of sulphurous oxide.
 Equation "b" 1 atom iodine equals $\frac{1}{4}$ molecule of arsenous oxide.
 Equation "c" 1 atom iodine equals $\frac{1}{2}$ atom of iron.
 Equation "d" 1 atom iodine equals 1 atom of bromine.
 Equation "e" 1 atom iodine equals 1 atom of chlorine.
 Equation "f" 1 atom iodine equals $\frac{1}{2}$ molecule of chromic oxide.
 Equation "g" 1 atom iodine equals 1 molecule of ferric chloride (or 1 atom of ferric iron).

Comparing these tabulated statements with equivalents for 1 Cc. $\frac{1}{10}$ n iodine solution and $\frac{1}{10}$ n thio solution given above, we find that the quantity of sulphurous oxide, arsenous oxide, and chromic acid represented by 1 Cc. $\frac{1}{10}$ n iodine or $\frac{1}{10}$ n thio is, respectively, one-half, one-quarter, and one-third the molecular weight expressed in tenths of milligrammes; while the quantity of ferrous iron, bromine, chlorine, and ferric iron is respectively one-half, one, one and one times the atomic weight expressed in tenths of milligrammes.

OFFICIAL DIRECT ASSAYS WITH TENTH-NORMAL SODIUM THIOSULPHATE V.S.

Iodine.—Place about 0.5 Gm. of Iodine in a tightly stoppered weighing-bottle and weigh accurately; add 1 Gm. of potassium iodide and dissolve in 50 Cc. of water, then add tenth-normal sodium thiosulphate V.S. until the liquid is decolorized. The number of Cc. of tenth-normal sodium thiosulphate T.S. consumed, when multiplied by 1.259, and divided by the weight of the Iodine taken, gives the percentage of pure Iodine present.

Tincture of Iodine.—If 5 Cc. of the Tincture be mixed with about 25 Cc. of water and titrated with tenth-normal sodium thiosulphate V.S., about 27.25 Cc. of tenth-normal sodium thiosulphate V.S. should be required for complete decolorization (corresponding to about 6.86 Gm. of Iodine in 100 Cc.).

Like assay of tincture of iodine are the assays for—

Compound Solution of Iodine.—6.3 Gm. takes 24.75 Cc. $\frac{1}{10}$ n thio to decolorize.

Sulphur Iodide.—0.5 Gm. substance, 1 Gm. potassium iodide, 25 Cc. water, take 28.00 Cc. $\frac{1}{10}$ n thio to decolorize, starch paste being used as indicator.

OFFICIAL DIRECT ASSAY WITH TENTH-NORMAL IODINE V.S.

Sodium Thiosulphate.—If 1 Gm. of Sodium Thiosulphate be dissolved in 20 Cc. of water, it should require the addition of not less than 39.75 Cc. of tenth-normal iodine V.S. to produce a slight yellow tint.

OFFICIAL REDUCTION ASSAYS WITH TENTH-NORMAL IODINE V.S.

Sulphurous Acid.—Introduce into a stoppered weighing-bottle 2 Cc. of Sulphurous Acid and weigh accurately. To this add 50 Cc. of tenth-normal iodine V.S., and allow it to stand for five minutes, then slowly add tenth-normal sodium thiosulphate V.S. until the mixture is just decolorized. Subtract the number of cubic centimeters of the tenth-normal sodium thiosulphate V.S. used from 50, and multiply the difference by 0.318, and divide this product by the weight of the Acid taken; the quotient represents the percentage of absolute Sulphurous Acid in the latter.

Sodium Bisulphite.—If to 50 Cc. of tenth-normal iodine V.S., measured from a burette into a glass-stoppered vial (of about 100-Cc. capacity), 0.25 Gm. of finely powdered crystals of Sodium Bisulphite be added, the solution allowed to stand for about an hour, and shaken at frequent intervals, then the addition of not more than 6.45 Cc. of tenth-normal sodium thiosulphate V.S. should be required to decolorize the solution.

Sodium Sulphite, like assay for sodium bisulphite, except 0.5 Gm. substance plus 50 Cc. $\frac{1}{10}$ N iodine solution takes 11.65 Cc. $\frac{1}{10}$ N thio solution to decolorize.

Reduced Iron.—Introduce about 2.6 Gm. of iodine into a 100-Cc. flask and weigh accurately, then add 6 Cc. of water, 2 Gm. of potassium iodide, and 0.555 Gm. of Reduced Iron. Securely stopper the flask, and, after thoroughly mixing the contents by rotating the flask, set it aside for one hour. Then dilute the contents with sufficient distilled water to make the liquid measure exactly 100 Cc., mix well, and to 25 Cc. of this solution slowly add tenth-normal sodium thiosulphate V.S., with constant stirring, until the last trace of brown color has been discharged. Divide the weight of iodine taken by 0.02518, and subtract from the quotient twice the number of Cc. of tenth-normal sodium thiosulphate V.S. used; the remainder represents the percentage of metallic iron present in the Reduced Iron, and this should not be less than 90 per cent.

NOTE.—The percentage purity of the iodine employed should be accurately determined by a previous experiment, and in place of the 2.6 Gm. above directed, its equivalent in pure (100 per cent.) iodine may be taken.

Arsenic Trioxide.—If 0.1 Gm. of Arsenic Trioxide be dissolved, together with 1 Gm. of sodium bicarbonate, in 20 Cc. of water, by the aid of a gentle heat, it should decolorize not less than 20.3 (20.32) Cc. of tenth-normal iodine V.S. (corresponding to at least 99.8 per cent. of pure Arsenic Trioxide).

Solution of Arsenous Acid.—See p. 183.

Solution of Potassium Arsenite.—If 24.6 Gm. of Solution of Potassium Arsenite be diluted with water to 100 Cc., the mixture very slightly acidified with diluted hydrochloric acid, and then made alkaline with 2 Gm. of sodium bicarbonate, it should require not less than 50 Cc. of tenth-normal iodine V.S. to produce a permanent yellow tint (corresponding to 1 Gm. of arsenic trioxide in 100 Gm. of the Solution).

Arsenous Iodide.—If 0.5 Gm. of Arsenous Iodide and 2 Gm. of sodium bicarbonate be dissolved in 50 Cc. of water, not less than 21.9 Cc. of tenth-normal iodine V.S. should be required to impart a slight yellow tint to the solution.

Antimony and Potassium Tartrate.—If 1 Gm. of Antimony and Potassium Tartrate be dissolved in sufficient water to measure 100 Cc., then 33 Cc. (32.99 Cc.) of this solution should, after the addition of 20 Cc. of a cold saturated aqueous solution of sodium bicarbonate and a little starch T.S., require not less than 19.9 Cc. of tenth-normal iodine V.S. to produce a permanent blue color (each Cc. corresponding to 5 per cent. of the pure salt). Titration should begin immediately after the addition of the sodium bicarbonate solution.

OFFICIAL OXIDATION ASSAYS WITH TENTH-NORMAL SODIUM THIOSULPHATE V.S.

Chlorinated Lime.—Introduce into a stoppered weighing-bottle between 3 and 4 Gm. of Chlorinated Lime and weigh accurately; triturate this thoroughly with 50 Cc. of water, transfer the mixture to a graduated vessel, together with the rinsings, and add sufficient water to make 1000 Cc. After thoroughly shaking, add to 100 Cc. of the mixture 1 Gm. of potassium iodide, 5 Cc. of diluted hydrochloric acid, and sufficient tenth-normal sodium thiosulphate V.S. for complete decolorization. Multi-

ply the number of Cc. of tenth-normal sodium thiosulphate V.S. consumed by 0.3518, and divide this product by one-tenth of the weight of the Chlorinated Lime taken; the quotient represents the percentage of available chlorine present.

Solution of Chlorinated Soda.—If 7 Gm. of the Solution be mixed with 50 Cc. of water, and 2 Gm. of potassium iodide and 10 Cc. of hydrochloric acid be added, not less than 48 Cc. of tenth-normal sodium thiosulphate V.S. should be required to discharge the final yellow color of the liquid (each Cc. of the tenth-normal sodium thiosulphate V.S. corresponding to 0.05 per cent. of available chlorine).

Chromium Trioxide.—If 1 Gm. of Chromium Trioxide be dissolved in 100 Cc. of water, then 8.3 (8.28) Cc. of this solution, when measured into a glass-stoppered bottle (of about 200-Cc. capacity), mixed with 2 Cc. of hydrochloric acid and about 1 Gm. of potassium iodide, after securely closing and shaking for a few minutes, should require, after diluting with 100 Cc. of water and adding 5 Cc. of starch T.S., not less than 22.5 Cc. of tenth-normal sodium thiosulphate V.S. to change the deep blue color to a light green (each Cc. of the tenth-normal sodium thiosulphate V.S. indicating 4 per cent. of pure Chromium Trioxide).

Ferric Chloride.—If 1 Gm. of dry Ferric Chloride be dissolved in sufficient water to measure 100 Cc., then 55.5 Cc. of this solution, when measured into a glass-stoppered flask of the capacity of about 250 Cc., followed by 3 Cc. of hydrochloric acid and 2 Gm. of potassium iodide, should, after securely stoppering the flask and heating for half an hour at 40° C. (104° F.), and cooling, require not less than 22 Cc. of tenth-normal sodium thiosulphate V.S. for complete decolorization (each Cc. of the tenth-normal sodium thiosulphate V.S. indicating 1 per cent. of metallic iron).

Similar to the assay for ferric chloride are the assays for the other official ferric salts, the differences being presented below in tabulated form:

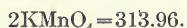
IRON SALT.	AMOUNT SUBSTANCE USED IN ASSAY.	AMOUNT WATER USED.	AMOUNT HCl USED.	AMOUNT KI USED.	STARCH PASTE USED.	Cc. $\frac{1}{10}$ N THIO V.S. REQUIRED TO DECOLOR- IZE.
<i>Solution of ferric chloride.</i>	11.1 Cc. of a dilution of 10 Gm. to water q.s. 100 Cc.	10 Cc.	2 Cc.	1 Gm.	Few drops.	At least 20 Cc.
<i>Tincture of ferric chloride.</i>	Evaporate 2.22 Gm. to dryness, add 2 Cc. hydrochloric acid and 5 Cc. solution of hydrogen dioxide, evaporate, and dissolve residue in water 10 Cc.	10 Cc.	1 Cc.	1 Gm.	"Little."	At least 18.3 Cc.
<i>Ferric ammonium sulphate.</i>	0.555 Gm.	15 Cc.	2 Cc.	1 Gm.	None.	At least 11.5 Cc.
<i>Solution of ferric sulphate.</i>	1.11 Gm.	15 Cc.	2 Cc.	1 Gm.	Few drops.	At least 20 Cc.
<i>Solution of ferric subsulphate.</i>	11.1 Cc. of a dilution of 10 Gm. to water q.s. 100 Cc.	10 Cc.	2 Cc.	1 Gm.	Few drops.	At least 27.2 Cc.
<i>Iron and ammonium tartrate.</i>	0.555 Gm.	15 Cc.	2 Cc.	1 Gm.	Yes.	At least 13 Cc.
<i>Iron and potassium tartrate.</i>	0.555 Gm.	15 Cc.	2 Cc.	1 Gm.	Yes.	At least 15 Cc.
<i>Ferric citrate.</i>	0.555 Gm.	15 Cc.	2 Cc.	1 Gm.	Yes.	At least 16 Cc.
<i>Iron and ammonium citrate.</i>	0.555 Gm.	15 Cc.	2 Cc.	1 Gm.	Yes.	At least 16 Cc.
<i>Iron and quinine citrate.</i>	Remove quinine from 1.11 Gm. substance by dissolving in 20 Cc. water, adding 5 Cc. ammonia water, and shaking out with chloroform; heat residual liquid on water-bath to remove last traces of chloroform and ammonia; dilute to 50 Cc. and use 25 Cc. of the dilution.		3 Cc.	1 Gm.	Yes.	At least 13.5 Cc.
<i>Soluble iron and quinine citrate.</i>	Prepared like iron and quinine citrate, only start with 4.44 Gm. substance, dilute to 100 Cc., and use 25 Cc. of the dilution.		4 Cc.	1 Gm.	Yes.	At least 32 Cc.
<i>Soluble ferric phosphate.</i>	0.555 Gm.	10 Cc.	2 Cc.	1 Gm.	No.	At least 12 Cc.
<i>Soluble ferric pyrophosphate.</i>	0.555 Gm.	10 Cc.	2 Cc.	1 Gm.	No.	At least 10 Cc.

OXIDATION ASSAYS WITH TENTH-NORMAL POTASSIUM PERMANGANATE V.S.

We have already learned (p. 384) that potassium permanganate is a powerful oxidizer, and that a solution in the process of oxidation loses its characteristic purple or rose tint—becomes decolorized. This phenomenon, employed in the volumetric estimation of oxidizable substances, to known quantities of such substances being added volumetric solution of potassium permanganate until a permanent pink tint ensues. In other cases advantage is taken of the fact that oxalic acid decolorizes potassium permanganate, and by mixing chemicals that react with oxalic acid with a known quantity of volumetric solution of that acid and titrating the excess of the acid with permanganate, the quantity of acid used in combining with the particular chemical can be deduced and the amount of the chemical calculated.

Before discussing the arithmetic of such calculation it may be well to give the official method of making decinormal permanganate solution:

TENTH-NORMAL POTASSIUM PERMANGANATE VOLUMETRIC SOLUTION



3.1396 Gm.¹ in 1000 Cc.

Introduce about 3.3 Gm. of pure, crystallized potassium permanganate [*Potassii Permanganas*, U. S. P.] into a flask, add 1000 Cc. of distilled water, and boil for about five minutes. Close the flask with a plug of absorbent cotton, and set aside for at least two days, so that suspended matter may deposit. After the lapse of this time, pour off the clear portion of the solution into a glass-stoppered bottle.

The water to be employed for diluting this solution (which is still too concentrated) should be prepared as directed under Distilled Water [*Aqua Destillata*, U. S. P.], adding, however, about 1 Gm. of potassium permanganate to the water in the retort before beginning the distillation.

I. Introduce into a flask 10 Cc. of an accurately standardized tenth-normal oxalic acid V.S., add 1 Cc. of pure concentrated sulphuric acid, and before this mixture cools, gradually add, from a burette provided with a glass stop-cock, small quantities of the permanganate solution to be standardized, shaking the flask after each addition and reducing the flow to drops toward the end of the operation. When the last drop of the permanganate solution added is no longer decolorized, but imparts a pinkish tint to the liquid, which remains permanent for one-half minute, note the number of Cc. consumed, and then dilute the trial permanganate solution with the specially prepared distilled water so that it will correspond, volume for volume, at 25° C. (77° F.), with the tenth-normal oxalic acid V.S. (note example under II.).

II. Tenth-normal potassium permanganate V.S. may also be standardized as follows:

To a solution of about 1 Gm. of potassium iodide [*Potassii Iodidum*, U. S. P.], in 10 Cc. of diluted sulphuric acid, contained in a flask, add, from a burette provided with a glass stop-cock, 20 Cc. of the potassium permanganate solution to be standardized; then dilute the mixture at once with about 200 Cc. of distilled water. An accurately standardized tenth-normal sodium thiosulphate V.S. is then slowly added from a burette, while the mixture is vigorously shaken, until the color is discharged. Note the number of Cc. of the latter consumed, then dilute the permanganate solution so that equal volumes of the two solutions correspond to each other under the same conditions at 25° C. (77° F.).

EXAMPLE.—Assuming that 25 Cc. of the tenth-normal sodium thiosulphate V.S. were required to decolorize the liberated iodine of the mixture, then each 20 Cc. of the potassium permanganate solution must be diluted with the specially prepared distilled water to 25 Cc., or the whole of the remaining solution in the same proportion. Thus, if 920 Cc. remain, it should be diluted to measure 1.150 Cc. at 25° C. (77° F.).

After the potassium permanganate solution is thus diluted, a new trial should be made in the manner above described, in which 20 Cc. of this solution should require

¹ The exact quantity is never weighed, but the solution is adjusted directly by oxalic acid or indirectly with sodium thiosulphate.

exactly 20 Cc. of the tenth-normal sodium thiosulphate V.S. to decolorize the mixture. If necessary, a new adjustment should be made to render the correspondence perfect.

NOTE.—When potassium permanganate V.S. is to be prepared for immediate use, this may be done as follows: Dissolve about 3.3 Gm. of pure crystallized potassium permanganate in 1000 Cc. of recently boiled and cooled pure water. This is then standardized by either of the above methods and diluted accordingly with recently boiled and cooled pure water. Potassium permanganate V.S. made by this method without the preliminary boiling and standing deteriorates readily, hence should be verified each time it is used.

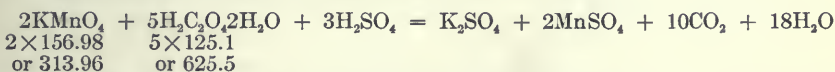
Potassium permanganate V.S. should be kept in well-closed glass-stoppered bottles, and only burettes provided with glass stop-cocks should be employed in titrating with it. Even when properly prepared and preserved, this solution should be restandardized frequently.

One Cubic Centimeter of Tenth-Normal Potassium Permanganate V.S. is the equivalent of:

	Gramme.
Potassium Permanganate, KMnO_4	0.0031396
Calcium Oxide, CaO (as oxalate).....	0.002784
Iron, Fe, in ferrous compounds.....	0.005550
Ferrous Carbonate, FeCO_3	0.011505
Ferrous Oxide, FeO	0.007138
Ferrous Sulphate, anhydrous, FeSO_4	0.015085
Ferrous Sulphate, crystals, $\text{FeSO}_4 + 7\text{H}_2\text{O}$	0.027601
Ferrous Sulphate, dried, $2\text{FeSO}_4 + 3\text{H}_2\text{O}$	0.017767
Hydrogen Dioxide, H_2O_2	0.001688
Oxalic Acid, crystallized, $\text{H}_2\text{C}_2\text{O}_4 + 2\text{H}_2\text{O}$	0.006255
Oxygen, O.....	0.000794
Sodium Nitrite, NaNO_2	0.0034285

It will be seen from the above recipe that the potassium permanganate solution is standardized by two methods—the one, the oxalic acid process outlined above; the other, by utilizing the property, possessed by permanganate in common with all other oxidizing agents, of liberating iodine from potassium iodide, estimating the amount of freed iodine with sodium thiosulphate solution. As this latter process has just been explained (p. 1000), we will proceed forthwith to the estimation of permanganate with oxalic acid.

The reaction of this combination is as follows:



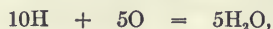
It will be seen from this equation that 625.5 Gm. oxalic acid decolorizes 313.96 Gm. potassium permanganate, and since 1 liter (1000 Cc.) decinormal oxalic acid contains 6.225 Gm. of the acid, then a liter of decinormal permanganate solution must contain

$$\frac{6.225}{625.500} \times 313.96, \text{ or } 3.1396 \text{ Gm. absolute permanganate.}$$

At first glance it seems erroneous to call the permanganate solution containing in a liter the number of grammes equivalent to one-fiftieth its molecular weight a decinormal solution, and it might here be stated that this conception is hard to comprehend unless we adopt the double formula of permanganate, $\text{K}_2\text{Mn}_2\text{O}_8$. If we take this double formula to express one molecule; if we appreciate that $\text{K}_2\text{Mn}_2\text{O}_8$ liberates 5 atoms oxygen in oxidation, viz.,



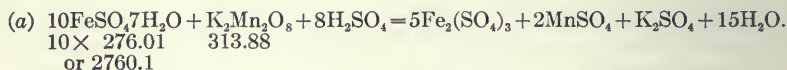
if we appreciate that this liberation of five atoms of oxygen really means combination of ten atoms of hydrogen, viz.,



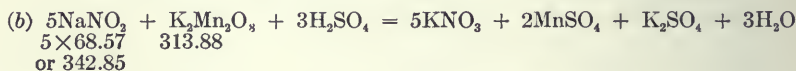
and, lastly, if we remember that the foundation of the arithmetic of volumetric testing is the quantity of substance that combines with or replaces one atom of hydrogen, we will see that the true normal solution of permanganate contains the amount with which one atom of hydrogen will combine—that is, $\frac{1}{10}$ of 313.96—and that the decinormal solution must contain in 1 liter $\frac{1}{100}$ of 313.96 Gm.

That this reasoning is sound is proved by the fact that a decinormal solution containing 3.13 Gm. permanganate to the liter combines with equal volumes of other decinormal solutions, such as $\frac{1}{10}$ n oxalic acid V.S., and in the second process of standardization, $\frac{1}{10}$ n thiosulphate V.S.

Referring to the table given above, showing the equivalent values of one cubic centimeter of $\frac{1}{10}$ n permanganate, we find that while $\frac{1}{10}$ n $\text{K}_2\text{Mn}_2\text{O}_8$ equals as many milligrammes of permanganate as its molecular weight divided by 100, 1 Cc. represents of the ferrous salts and hydrogen dioxide as many milligrammes as the molecular weight in each case divided by 10; while with the substances, calcium oxide, oxalic acid, and sodium nitrite, 1 Cc. of $\frac{1}{10}$ n permanganate represents as many milligrammes as the molecular weight divided by 20. These can be best explained by a typical equation from each class:



We see that 313.88 Gm. $\text{KMnO}_4 = 2760.1$ Gm. $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$.
 3.1388 Gm. KMnO_4
 or 1000 Cc. $\frac{1}{10}$ n $\text{KMnO}_4 = 27.601$ Gm. $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$.
 and that 1 Cc. $\frac{1}{10}$ n $\text{KMnO}_4 = 0.0276$ Gm. $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$.



We see that 313.88 Gm. $\text{KMnO}_4 = 342.85$ Gm. NaNO_2
 3.1388 Gm. KMnO_4
 or 1000 Cc. $\frac{1}{10}$ n $\text{KMnO}_4 = 3.4285$ Gm. NaNO_2
 and that 1 Cc. $\frac{1}{10}$ n $\text{KMnO}_4 = 0.0034285$ Gm. NaNO_2 .

That is, while chemicals of the type "a" in oxidizing take up one atom of oxygen to every two molecules of the oxidized chemical, chemicals of type "b" combine with oxygen in the proportion of one atom of oxygen to each molecule.

It will, of course, be remembered that one molecule $\text{K}_2\text{Mn}_2\text{O}_8$ yields five atoms of oxygen, and these five atoms combine with ten molecules of chemicals of type "a" and with five molecules of chemicals of type "b."

OFFICIAL ASSAYS MADE WITH TENTH-NORMAL POTASSIUM PERMANGANATE V. S.

Solution of Hydrogen Dioxide.—Dilute 10 Cc. of the Solution with sufficient distilled water to measure 100 Cc. Transfer 16.9 Cc. of this liquid (containing 1.69 Cc.

of the Solution) to a beaker, add 5 Cc. of diluted sulphuric acid, and then, from a burette, slowly add, with constant stirring, tenth-normal potassium permanganate V.S. until the liquid just retains a faint pink tint. Each Cc. of tenth-normal potassium permanganate V.S. consumed corresponds to 0.1 per cent. of absolute Hydrogen Dioxide or 0.329 volume of oxygen. If the Solution be of full strength, 30 Cc. of tenth-normal potassium permanganate V.S. will be required.

This reaction differs from either equation given on the previous page, inasmuch as both hydrogen dioxide and permanganate liberate oxygen, which passes off as gas, as shown in the following equation:



Ferrous Sulphate.—If 1.38 Gm. of the salt, in uneffloresced crystals, be dissolved in about 25 Cc. of diluted sulphuric acid, not less than 49.75 Cc. of tenth-normal potassium permanganate V.S. should be required to impart to the liquid a permanent pink color (each Cc. of the tenth-normal potassium permanganate V.S. indicating 2 per cent. of crystallized Ferrous Sulphate).

In the two following assays oxalic acid acts on the chemical and the excess of oxalic acid is then estimated with permanganate:

Precipitated Manganese Dioxide.—If 0.2 Gm. of Precipitated Manganese Dioxide be dissolved in a mixture of 50 Cc. of tenth-normal oxalic acid V.S. and 3 Cc. of sulphuric acid contained in a flask and heated on a water-bath, the resulting solution, after dilution with 100 Cc. of warm water, should require the addition of not more than 13 (12.95) Cc. of tenth-normal potassium permanganate V.S. to produce a slight pink tint (corresponding to not less than 80 per cent. of pure Manganese Dioxide).

Solution of Lead Subacetate.—If 10 Gm. of the Solution be diluted with distilled water, which has been previously boiled and cooled, to measure 100 Cc., and 13.6 (13.594) Cc. of this be added to 35 Cc. of tenth-normal oxalic acid V.S., contained in a graduated cylinder, and, after thoroughly shaking, the mixture be diluted with distilled water to measure 50 Cc., and again well shaken, then, after the precipitate has settled, 10 Cc. of the clear solution, after diluting with about 50 Cc. of water and adding 5 Cc. of sulphuric acid, should require not more than 2 Cc. of tenth-normal potassium permanganate V.S. to produce a permanent pink tint (each Cc. of tenth-normal oxalic acid V.S. required for the precipitation of the 13.6 Cc. of the diluted Solution corresponding to 1 per cent. of Lead Subacetate).

In the following assays the titration is with tenth-normal oxalic acid V.S. until the pink color of the permanganate is changed:

Sodium Nitrite.—If to 30 Cc. of tenth-normal potassium permanganate V.S., diluted with about 150 Cc. of distilled water, 5 Cc. of sulphuric acid and 10 Cc. of a solution of 1 Gm. of Sodium Nitrite in sufficient distilled water to make 100 Cc. be successively added, the liquid brought to a temperature of 40° C. (104° F.), and allowed to stand for five minutes, not more than 3.75 Cc. of tenth-normal oxalic acid V.S. should be required to decolorize the solution (each Cc. of tenth-normal potassium permanganate consumed corresponding to 0.0034285 Gm. of pure Sodium Nitrite).

Potassium Permanganate.—If 0.1 Gm. of Potassium Permanganate be dissolved in about 100 Cc. of hot distilled water, to which 1 Cc. of sulphuric acid has previously been added, the solution should require for complete decolorization not less than 31.5 Cc. of tenth-normal oxalic acid V.S.

OXIDATION ASSAYS WITH TENTH-NORMAL POTASSIUM DICHROMATE V.S.

As the operation is so closely similar to assaying with potassium permanganate, and as only one official chemical is directed to be tested by this method, we need not take much time for its discussion.

All that is necessary to state is that the oxidation with dichromate

is limited to ferrous salts and to the liberation of iodine from potassium iodide in standardizing sodium thiosulphate V.S. Further, that the oxidizing action of dichromate is that shown in the following typical equation:



In other words, while *two* molecules nitric acid in oxidation processes give three atoms of oxygen (see p. 420), one molecule dichromate gives three atoms of oxygen also. Hence when dichromate can be substituted for nitric acid in oxidations, one molecule does the work of two molecules of acid.

Since one molecule $\text{K}_2\text{Cr}_2\text{O}_7$ yields three atoms of oxygen, one molecule $\text{K}_2\text{Cr}_2\text{O}_7$ is the equivalent of six atoms of hydrogen; hence normal potassium dichromate solution would contain in 1000 Cc. as many grammes $\text{K}_2\text{Cr}_2\text{O}_7$ as the molecular weight (292.28) divided by 6: that is, 48.713 Gm. Therefore decinormal dichromate V.S. contains 4.8713 Gm. $\text{K}_2\text{Cr}_2\text{O}_7$ to the liter, as is shown in the pharmacopœial recipe given below.

TENTH-NORMAL POTASSIUM DICHROMATE VOLUMETRIC SOLUTION

$\text{K}_2\text{Cr}_2\text{O}_7 = 292.28.$

4.8713 Gm. in 1000 Cc.

Dissolve 4.8713 Gm. of pure potassium dichromate, which has been pulverized and dried at 120°C. (248°F.), in sufficient water to measure, at 25°C. (77°F.), exactly 1000 Cc.

When used with phenolphthalein as indicator to neutralize alkalis, the volumetric solution of potassium dichromate is tenth-normal when it contains 14.614 Gm. in 1000 Cc. It is then the exact equivalent of any tenth-normal acid V.S., each Cc. being equivalent to the amounts of alkalis quoted under such acids.

When used as an oxidizing agent to convert ferrous into ferric salts, or to liberate iodine from potassium iodide, the solution just mentioned (containing 14.614 Gm. in 1000 Cc.) has the effect of a $\frac{3\text{N}}{10}$ volumetric solution, and a solution of one-third of this strength, containing 4.8713 Gm. in 1000 Cc., has the value of a tenth-normal solution, and is the equivalent of an equal volume of tenth-normal potassium permanganate V.S., or, in the case of iodine liberated from potassium iodide, it is the equivalent of an equal volume of tenth-normal sodium thiosulphate V.S. For titrating iron in *ferrous* compounds, it is used in the following manner: Introduce the aqueous solution of the ferrous salt into a flask and, if it is not already acid, render it so with sulphuric acid. Now add, gradually, tenth-normal potassium dichromate V.S. from a burette, with agitation, until a drop taken out upon a white surface no longer becomes blue when mixed with a drop of freshly prepared potassium ferricyanide T.S.

Tenth-normal potassium dichromate V.S. may also be used, in conjunction with potassium iodide (from which it liberates iodine) and sulphuric acid, for adjusting the titer of sodium thiosulphate V.S. and thus that of the iodine V.S.

One Cubic Centimeter of Tenth-Normal Potassium Dichromate V.S. is the equivalent of:

	Gramme.
Potassium Dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$	0.0048713
Iron, Fe, in ferrous compounds.....	0.00555
Ferrous Carbonate, FeCO_3	0.011505
Ferrous Sulphate, anhydrous, FeSO_4	0.015085
Ferrous Sulphate, crystallized, $\text{FeSO}_4 + 7\text{H}_2\text{O}$	0.027601
Ferrous Sulphate, dried, $2\text{FeSO}_4 + 3\text{H}_2\text{O}$	0.017767
Sodium Thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$	0.024646

OFFICIAL ASSAY MADE WITH TENTH-NORMAL POTASSIUM DICHROMATE V.S.

Saccharated Ferrous Carbonate.—If 1.15 Gm. of Saccharated Ferrous Carbonate be dissolved in 10 Cc. of diluted sulphuric acid (1 to 5), and the solution diluted with water to about 100 Cc., it should require not less than 15 Cc. of tenth-normal potassium dichromate V.S. for complete oxidation, potassium ferricyanide T.S. being used as indicator (corresponding to not less than 15 per cent. of ferrous carbonate).

VOLUMETRIC ASSAYS WITH SILVER NITRATE, POTASSIUM SULPHOCYANATE, AND SODIUM CHLORIDE

This class of assays is based either on the ability of silver nitrate to precipitate in definite proportions with the halogens, as shown in the following equation:



or with potassium sulphocyanate, viz.,



From the above equations and appended molecular weights we see that 168.69 Gm. $\text{AgNO}_3 = 58.06$ Gm. NaCl , and that 168.69 Gm. $\text{AgNO}_3 = 96.53$ Gm. KSCN .

Since $\frac{1}{10}$ n AgNO_3 V.S. contains 16.869 Gm. AgNO_3 to 1000 Cc., and $\frac{1}{10}$ n AgNO_3 V.S. contains 0.016869 Gm. AgNO_3 to 1 Cc., therefore 1 Cc. $\frac{1}{10}$ n AgNO_3 V.S. = 0.005806 Gm. NaCl and 1 Cc. $\frac{1}{10}$ n AgNO_3 V.S. = 0.009653 Gm. KSCN .

As fully explained below, the indicator used when assaying halogens with silver nitrate is potassium chromate, it striking a red, permanent tint (silver chromate) only after the addition of just enough silver nitrate completely to precipitate the halogen. When testing with sulphocyanate, ferric alum is used as indicator, it forming red ferric sulphocyanate only after all the silver is precipitated.

The preparation of the three volumetric solutions is given below:

TENTH-NORMAL SILVER NITRATE VOLUMETRIC SOLUTION

$\text{AgNO}_3 = 168.69.$ 16.869 Gm. in 1000 Cc.

Dissolve 16.869 Gm. of silver nitrate [*Argenti Nitras*, U. S. P.], which, previous to weighing, has been pulverized and dried in a covered porcelain crucible in an air-bath at 130°C . (266°F .) for one hour, in sufficient water to measure, at 25°C . (77°F .), exactly 1000 Cc.

Keep the solution in dark amber-colored, glass-stoppered vials, carefully protected from dust and sunlight.

NOTE.—Titration by tenth-normal silver nitrate V.S. may be conducted in various ways, adapted to the special preparation to be tested:

a. *The Titration of Soluble Chlorides and Bromides.*—To the solution of an accurately weighed quantity of the salt, contained in either a porcelain dish or a flask placed on a white surface, sufficient potassium chromate T.S. is added to impart a yellow tint, then the tenth-normal silver nitrate V.S. is slowly added from a burette, stirring or agitating constantly until the mixture acquires a permanent red tint, due to the formation of red silver chromate. This method is only suitable for *neutral* solutions.

b. *The Titration of Free Hydrobromic, Hydrochloric, and Hydriodic Acids or their Salts in Acid Solution, Known as the Volhard or Thiocyanate (Sulphocyanate) Method of Residual Titration.*—An accurately measured excess of tenth-normal silver nitrate V.S. is added to the solution of the halogen acid or its salt, and then acidified with pure

nitric acid, and the indicator (ferric ammonium sulphate T.S.) added; then the uncombined excess of the silver nitrate V.S. is determined by titrating back with tenth-normal potassium sulphocyanate V.S., the end-reaction being the formation of a permanent pale reddish-brown tint (due to ferric sulphocyanate). The volume of the silver nitrate V.S. originally added, less that of the potassium sulphocyanate V.S. consumed, will give the number of Cc. of the former which were required for the precipitation of the halogen. The quantity of nitric acid added should be sufficient to remove the yellow color produced by the addition of the indicator.

c. *Titration Till the First Appearance of a Permanent Precipitate.*—This method is applicable in the estimation of the alkali cyanides and hydrocyanic acid. When the solution is used by this method it is fifth-normal instead of tenth-normal solution.

One Cubic Centimeter of Tenth-Normal Silver Nitrate V.S. is the equivalent of:

	Gramme.
Silver Nitrate, AgNO_3	0.016869
Allyl Iso-thiocyanate, $\text{CS.NC}_3\text{H}_5$	0.00492
Ammonium Bromide, NH_4Br	0.009729
Ammonium Chloride, NH_4Cl	0.005311
Ammonium Iodide, NH_4I	0.014383
Bromine, Br.....	0.007936
Calcium Bromide, CaBr_2	0.009926
Chlorine, Cl.....	0.003518
Ferrous Bromide, anhydrous, FeBr_2	0.010711
Ferrous Iodide, FeI_2	0.015365
Hydriodic Acid, HI.....	0.01269
Hydrobromic Acid, HBr.....	0.008036
Hydrochloric Acid, HCl.....	0.003618
Hydrocyanic Acid, HCN, to first formation of precipitate.....	0.005368
Hydrocyanic Acid, HCN, potassium chromate as indicator.....	0.002684
Iodine, I.....	0.01259
Lithium Bromide, LiBr.....	0.008634
Potassium Bromide, KBr.....	0.011822
Potassium Chloride, KCl.....	0.007404
Potassium Cyanide, KCN, to first formation of precipitate.....	0.01294
Potassium Iodide, KI.....	0.016476
Potassium Sulphocyanate, KSCN.....	0.009653
Sodium Bromide, NaBr.....	0.010224
Sodium Chloride, NaCl.....	0.005806
Sodium Iodide, NaI.....	0.014878
Strontium Bromide, $\text{SrBr}_2 + 6\text{H}_2\text{O}$	0.017647
Strontium Iodide, $\text{SrI}_2 + 6\text{H}_2\text{O}$	0.022301
Zinc Bromide, ZnBr_2	0.011181
Zinc Chloride, ZnCl_2	0.006763
Zinc Iodide, ZnI_2	0.015835

TENTH-NORMAL SODIUM CHLORIDE VOLUMETRIC SOLUTION

$\text{NaCl} = 58.06$.

5.806 Gm. in 1000 Cc.

Dissolve 5.806 Gm. of pure sodium chloride (see below) in sufficient water to measure, at 25°C . (77°F .), exactly 1000 Cc.

Pure Sodium Chloride may be prepared by passing a current of dry hydrochloric acid gas into a saturated aqueous solution of the purest commercial sodium chloride, collecting the crystalline precipitate on a filter, washing with a little pure concentrated hydrochloric acid, draining, pulverizing, and igniting it gently in a crucible heated to low redness, to expel all traces of free acid. Care should be taken to avoid fusion.

One Cubic Centimeter of Tenth-Normal Sodium Chloride V.S. is the equivalent of:

	Gramme.
Sodium Chloride, NaCl.....	0.005806
Silver, Ag.....	0.010712
Silver Nitrate, AgNO_3	0.016869
Silver Oxide, Ag_2O	0.011506

TENTH-NORMAL POTASSIUM SULPHOCYANATE VOLUMETRIC SOLUTION

[VOLHARD'S SOLUTION]

KSCN = 96.53.

9.653 Gm. in 1000 Cc.

Dissolve 10 Gm. of crystals of pure potassium sulphocyanate in 1000 Cc. of water.

This solution is too concentrated, and has to be adjusted so as to correspond in strength exactly with tenth-normal silver nitrate V.S. For this purpose, introduce into a flask 10 Cc. of tenth-normal silver nitrate V.S., 3 Cc. of ferric ammonium sulphate T.S., and 3 Cc. of nitric acid (free from nitrous compounds), and dilute the liquid with about 100 Cc. of distilled water. To this mixture add, from a burette, in small portions at a time, the sulphocyanate solution. At first, a white precipitate of silver sulphocyanate appears, then, every drop falling from the burette is surrounded by a deep brownish-red color produced by ferric sulphocyanate, which disappears on vigorous shaking of the flask as long as any of the silver nitrate remains unchanged. When all the silver has been converted into sulphocyanate, a single additional drop of the potassium sulphocyanate solution produces a brownish-red color which no longer disappears on shaking, but communicates a perceptible pale reddish-brown tint to the contents of the flask. Note the number of Cc. of the potassium sulphocyanate solution used, and dilute the whole of the remaining solution so that equal volumes of this and of the tenth-normal silver-nitrate V.S., at 25° C. (77° F.), will be required to produce the permanent reddish-brown tint. (The same depth of pale reddish-brown tint to which the volumetric solution is adjusted must be attained when the solution is used for volumetric assays.)

After the dilution, a new trial should be made, in which 50 Cc. of tenth-normal silver nitrate V.S., 5 Cc. of ferric ammonium sulphate T.S., 5 Cc. of nitric acid, and 200 Cc. of water are used, and there should be required exactly 50 Cc. of the potassium sulphocyanate solution, at 25° C. (77° F.), to produce the same depth of a permanent pale reddish-brown tint.

If necessary, a new adjustment should be made to render the correspondence perfect.

One Cubic Centimeter of Tenth-Normal Potassium Sulphocyanate V.S. is the equivalent of:

	Gramme.
Potassium Sulphocyanate, KSCN.....	0.009653
Silver, Ag.....	0.010712
Silver Nitrate, AgNO ₃	0.016869

OFFICIAL ASSAYS WITH TENTH-NORMAL SILVER NITRATE V.S.

Diluted Hydrobromic Acid.—If 10 Gm. of Diluted Hydrobromic Acid be diluted with sufficient distilled water to measure 100 Cc., and if to 8.04 Cc. of this solution after exact neutralization with diluted ammonia water (litmus T.S. being used as indicator), there be added 3 drops of potassium chromate T.S., then not less than 10 Cc. of tenth-normal silver nitrate V.S. should be required to impart to the liquid a permanent red tint (each Cc. corresponding to 1 per cent. of absolute Hydrobromic Acid).

Potassium Bromide.—If 0.3 Gm. of the well-dried salt be dissolved in about 50 Cc. of water, and 2 or 3 drops of potassium chromate T.S. be added, it should require not less than 24.6 Cc. nor more than 25.85 Cc. of tenth-normal silver nitrate T.S. to produce a permanent red color.

Taking the assay of this well-known chemical as a type, we see by reference to the equivalent table, given on p. 1012, that 1 Cc. tenth-normal AgNO₃ V.S. = 0.011822 Gm. KBr.

Then 25 Cc. tenth-normal AgNO₃ V.S. = $25 \times 0.011822 = 0.295550$ Gm. KBr.

If 0.3 Gm. KBr took 25 Cc. tenth-normal AgNO_3 V.S., then 0.3 Gm. KBr contained 0.29555 Gm. absolute KBr., and the strength of such potassium bromide is $\frac{29555}{30000} \times 100$ per cent., or 98.5 per cent. absolute KBr.

The following chemicals are assayed like potassium bromide, the differences in manipulation being presented in tabulated form:

CHEMICAL.	PREPARATION OF AQUEOUS SOLUTION.	AMOUNT OF K_2CrO_4 T.S. USED.	AMOUNT $\frac{1}{10}\text{N}\text{AgNO}_3$ V.S. NEEDED TO GIVE PERMANENT RED TINT.
<i>Potassium iodide.</i>	0.5 Gm. dissolved in 10 Cc. water.	3 drops.	Not less than 30 Cc.; not more than 30.8 Cc.
<i>Sodium bromide.</i>	0.3 Gm. dissolved in 50 Cc. water.	2 or 3 drops.	Not less than 25.8 Cc.; not more than 30 Cc.
<i>Sodium chloride.</i>	1 Gm. diluted to 100 Cc. 10 Cc. of this dilution taken.	Few drops.	Not less than 17 Cc.
<i>Sodium iodide.</i>	0.5 Gm. dissolved in 10 Cc. water.	5 drops.	Not less than 33 Cc.; not more than 34.6 Cc.
<i>Lithium bromide.</i>	1 Gm. diluted to 100 Cc. 20 Cc. of this dilution taken.	2 drops.	Not less than 22 Cc.; not more than 23.9 Cc.
<i>Ammonium bromide.</i>	3 Gm. diluted to 100 Cc. 10 Cc. of this dilution taken.	Few drops.	Not more than 31.6 Cc.
<i>Ammonium chloride.</i>	3 Gm. diluted to 100 Cc. 10 Cc. of this dilution taken.	Few drops.	Not less than 18.7 Cc.
<i>Strontium bromide.</i>	0.5 Gm. dissolved in 50 Cc. water.	Few drops.	Not less than 27.4 Cc.; not more than 29.4 Cc.
<i>Zinc bromide.</i>	0.3 Gm. dissolved in 10 Cc. water.	2 drops.	Not less than 26 Cc.; not more than 26.8 Cc.

Potassium Cyanide.—If 1 Gm. of Potassium Cyanide be dissolved in sufficient distilled water to measure 100 Cc., then 64.7 Cc. of this solution mixed with 5 Cc. of ammonia water and 3 drops of potassium iodide T.S. should require not less than 47.5 Cc. of tenth-normal silver nitrate V.S. before the appearance of a permanent precipitate (each Cc. of the tenth-normal silver nitrate V.S. indicating 2 per cent. of pure Potassium Cyanide).

This assay is based on the formation of a soluble double salt (AgCNKCN), which is decomposed the minute an excess of silver is added. Potassium iodide accentuates the reaction.

Similar assays are the following:

Diluted Hydrocyanic Acid.—If 5 Gm. of Diluted Hydrocyanic Acid be diluted with distilled water to measure 50 Cc., then 26.9 Cc. (26.84 Cc.) of this solution, after the addition of 5 Cc. of ammonia water and 3 drops of potassium iodide T.S., should require for the production of a slight permanent precipitate the addition of not less than 10 Cc. of tenth-normal silver nitrate V.S.

Oil of Bitter Almond.—Mix, in a 100-Cc. flask, 1 Gm. of the Oil of Bitter Almond to be tested, with sufficient water and freshly precipitated magnesium hydroxide (free from chlorides) to make an opaque mixture of about 50 Cc. Add to this 2 or 3 drops of potassium chromate T.S., and then from a burette add tenth-normal silver nitrate V.S. until a red tint is produced which does not again disappear by shaking; not less than 7.5 Cc. nor more than 14.9 Cc. of tenth-normal silver nitrate V.S. should be required, each Cc. corresponding to 0.002684 Gm. of hydrocyanic acid.

VOLUMETRIC ASSAYS WITH TENTH-NORMAL SILVER NITRATE V.S. AND TENTH-NORMAL SODIUM CHLORIDE V.S.

Silver Nitrate.—If 0.5 Gm. of Silver Nitrate, dissolved in 10 Cc. of distilled water, be well mixed with 30 Cc. of tenth-normal sodium chloride V.S. and 3 drops of potassium chromate T.S., not more than 0.4 Cc. of tenth-normal silver nitrate V.S. should be required to impart to the liquid a permanent red color.

In this assay an excess of sodium chloride V.S. is added and this excess titrated with tenth-normal silver nitrate V.S.

Explaining the assay arithmetically, we find, by reference to the equivalent tables on p. 1012:

$$1 \text{ Cc. } \frac{1}{10} \text{N NaCl} = 0.016869 \text{ Gm. AgNO}_3.$$

$$\text{Then } 30 \text{ Cc. } \frac{1}{10} \text{N NaCl} = 30 \times 0.016869 = 0.50607 \text{ Gm. absolute AgNO}_3.$$

We have to balance against this

$$\text{Silver nitrate weighed out} = 0.50000 \text{ Gm.}$$

$$\text{Silver nitrate contained in } 0.4 \text{ Cc. } \frac{1}{10} \text{N AgNO}_3 \text{ V.S.} = 0.006747 \text{ Gm.}$$

$$(0.4 \times 0.016869) \quad 0.506747 \text{ Gm.}$$

Hence the strength of the sample is

$$\frac{50607}{50674} \times 100 \text{ per cent.} = 99.8 \text{ per cent. absolute AgNO}_3.$$

In like manner are the following chemicals assayed:

Fused Silver Nitrate.—0.5 Gm. takes not more than 1.9 Cc. tenth-normal AgNO V.S. (Tenth-normal NaCl V.S., 20 Cc.; $\text{K}_2\text{Cr}_2\text{O}_7$ T.S., 3 drops.)

Mitigated Silver Nitrate.—1 Gm. takes not more than 0.3 Cc. tenth-normal AgNO_3 V.S. (Tenth-normal NaCl V.S., 20 Cc.; K_2CrO_4 T.S., 3 drops.)

VOLUMETRIC ASSAYS WITH TENTH-NORMAL SILVER NITRATE V.S. AND TENTH-NORMAL POTASSIUM SULPHOCYANATE V.S.

Diluted Hydriodic Acid.—Into a flask provided with a well-fitting stopper introduce 2.54 Gm. of Diluted Hydriodic Acid, diluted with 50 Cc. of distilled water, followed by 25 Cc. of tenth-normal silver nitrate V.S., 5 Cc. of ferric ammonium sulphate T.S., and 3 to 4 Cc. of nitric acid (free from nitrous compounds); then securely stopper the flask and shake it well. Not more than 5 Cc. of tenth-normal potassium sulphocyanate V.S. should then be required to produce a permanent reddish-brown tint (each Cc. of tenth-normal silver nitrate V.S. consumed corresponding to 0.5 per cent. of absolute Hydriodic Acid).

In this assay an excess of volumetric solution of silver nitrate is added, and after precipitation of silver iodide, the silver still remaining in solution is titrated with potassium sulphocyanate. The calculations whereby the figures given in the assay are obtained are as follows:

By reference to the equivalent tables given on p. 1012 find:

$$1 \text{ Cc. } \frac{1}{10} \text{N AgNO}_3 \text{ V.S.} = 0.01269 \text{ Gm. absolute HI,}$$

or, reversed,

$$0.01269 \text{ Gm. absolute HI takes } 1 \text{ Cc. } \frac{1}{10} \text{N AgNO}_3 \text{ V.S.}$$

$$\left. \begin{array}{l} \text{then } 0.254 \text{ Gm. absolute HI} \\ \text{or } 2.54 \text{ Gm. diluted HI} \end{array} \right\} \text{ take } \left\{ \begin{array}{l} .25400 \\ .01269 \end{array} \right. \times 1 \text{ Cc. } \frac{1}{10} \text{N AgNO}_3 \text{ V.S.}$$

$$\left. \begin{array}{l} .25400 \\ .01269 \end{array} \right\} \text{ or } 20.01 \text{ Cc. } \frac{1}{10} \text{N AgNO}_3 \text{ V.S.}$$

If we start in with 25 Cc. tenth-normal AgNO_3 V.S., and 20.01 Cc. of this is used by the hydriodic acid, there will remain in solution the silver equivalent of 5 Cc. tenth-normal AgNO_3 V.S. This will, of course, take 5 Cc. tenth-normal KSCN for exact combination.

In like manner are the following official chemicals assayed:

CHEMICAL.	PREPARATION OF AQUEOUS SOLUTION.	AMOUNT $\frac{1}{10}$ N AgNO_3 ADDED.	AMOUNT FERRIC ALUM T.S. ADDED.	AMOUNT NITRIC ACID ADDED.	AMOUNT $\frac{1}{10}$ N KSCN REQUIRED.
<i>Syrup of hydriodic acid.</i>	31.73 Gm. diluted to 50 Cc., and 10 Cc. of this plus 10 Cc. water taken.	8 Cc.	3 Cc.	5 Cc. diluted acid.	Not more than 3 Cc.
<i>Syrup of ferrous iodide.</i>	10 Gm. diluted to 100 Cc., and 15.4 Cc. of this plus 15 Cc. water taken.	6 Cc.	2 Cc.	2 Cc. diluted acid.	Not more than 1 Cc.
<i>Strontium iodide.</i>	0.5 Gm. in 100 Cc. water.	25 Cc.	5 Cc.	5 Cc. strong acid.	Not less than 1.7 Cc. nor more than 3.1 Cc.
<i>Zinc iodide.</i>	0.5 Gm. in 20 Cc. water.	35 Cc.	3 Cc.	5 Cc. strong acid.	Not less than 3.4 Cc. nor more than 4.0 Cc.

The following assay differs from the others only in the fact that the silver nitrate solution is used to combine with the alyl isothiocyanate of the oil, the excess of silver being titrated with sulphocyanate V.S.

Volatile Oil of Mustard.—Weigh accurately about 2 Gm. of Volatile Oil of Mustard and dilute this with sufficient alcohol to make 50 Cc. of the solution represent 1 Gm. of the Oil; of this solution, 5 Cc. are transferred to a 100-Cc. measuring flask, and 30 Cc. of tenth-normal silver nitrate V.S. and 5 Cc. of ammonia water are added. The flask is well stoppered and set aside in a dark place for twenty-four hours. The contents of the flask are diluted with water to the 100 Cc. mark and filtered. To 50 Cc. of the filtrate, 4 Cc. of nitric acid and a few drops of ferric ammonium sulphate T.S. are added, and finally sufficient tenth-normal potassium sulphocyanate V.S. to produce a permanent red color; not more than 5.6 Cc. of the latter reagent should be required (each Cc. of tenth-normal silver nitrate V.S. consumed corresponding to 0.00492 gramme of alyl iso-thiocyanate).

CHAPTER LV

VOLUMETRIC ASSAYS OF ORGANIC SUBSTANCES

PHARMACEUTIC ASSAYING

ASIDE from the volumetric estimation of organic acids described on p. 997 under Alkalimetry, there are several forms of volumetric assays peculiar to organic substances. These include estimation of saponification number and iodine absorption number of oils and fats; the acid number of resins; that modification of acidimetry applied in alkaloidal assays, as well as the special assay of sugars with Fehling's solution and of phenol with Koppeschaar's solution. These will be considered in this chapter.

ALKALOIDAL ASSAYS

As already outlined on p. 944, the official assays for alkaloids naturally fall into two groups—the isolation of the alkaloid and the estimation of the isolated alkaloid either by direct weighing or by estimation by titration with decinormal or centinormal acid (or indirectly by titrating excess of acid with volumetric alkali solution). As already mentioned, the alkaloid is isolated by extracting the drug with aqueous (either plain or acidulated) media, the alkaloid set free by addition of alkali, and the freed alkaloid separated by “shaking out” with a solvent immiscible with water. The pharmacopœial details of the “shaking out” process are given below.

ALKALOIDAL ASSAY BY IMMISCIBLE SOLVENTS

Nearly all alkaloids are practically insoluble in water, but they are soluble in alcohol, chloroform, ether, amyl alcohol, benzene, petroleum benzin, or mixtures of several of these. The salts of these alkaloids, however, are soluble in water, but practically insoluble in the above-mentioned solvents. The process of assay by immiscible solvents, which is generally known as the “shaking out” process, is based on this property of alkaloids, and it is carried out by treating liquid extracts that have been freed from alcohol, with an immiscible solvent in the presence of an excess of alkali. This liberates the alkaloid, and, on becoming free, if not so previously, it is dissolved by the immiscible solvent. This solution is then separated, transferred to another container, and shaken with an excess of acid largely diluted with water. The acid combining with the free alkaloid forms a salt, which now leaves the immiscible solvent and is found in the aqueous solution. This process is sometimes repeated, in case the alkaloidal solution is still colored. The apparatus used in this operation of shaking out is termed a “separator,” and consists of an oval or pear-shaped glass vessel with an opening at the top supplied with a well-ground glass stopper, and an outlet tube at the bottom, provided with an accurately fitting glass stop-cock (Fig. 140). The solvents directed to be used in this Pharmacopœia are alcohol, chloroform, ether, and various mixtures of both, containing at least 75 parts of ether in 100 parts of solvent by volume. In the case of chloroform, the solvent will collect at the bottom of the separator, and can be drawn off, but the ethereal or ether-chloroform mixture will form the upper portion of the liquid in the separator, and the aqueous layer must first be drawn off into a suitable vessel, and the ethereal layer then transferred to another vessel. It is not necessary or desirable to shake the mixture of immiscible solvent and water violently, for a rotation of the separator or a gentle shaking for about a minute will answer all purposes. At times, an emulsion of the water and the solvent is formed, especially if the shaking is too violent, and in order to separate this it is advisable to proceed as follows: If the solvent is heavier than the water, add more of the former, a little water, and a slight amount of alcohol; if the solvent is lighter than the water, add sufficient saturated sodium chloride solution or crystals of sodium chloride. A safe procedure to avoid the forming of emulsions is to invert the separator several times, and then to at once begin rotating to keep the solvents well mixed. To insure a complete extraction of the alkaloid, it is desirable to treat the liquid three times with the immiscible solvent, and this is to be followed by a rinsing of the empty separator with repeated small portions of the same solvent. The separator should not be filled to more than two-thirds of its capacity at any time, and if its contents should become heated by the neutralization of acid by alkali, or *vice versa*, it should be cooled to the temperature of the room, before opening the stopper, by immersing it in running water. The final operation must always be the collection of the free alkaloid by the use of a portion of the immiscible solvent, drawing this off into a beaker, rinsing with small portions of the solvent to prevent possible loss. The beaker is then placed on a water-bath and gently heated, to remove the solvent by evaporation, leaving the alkaloids in the beaker in the dry form, and usually in the condition of a resinous or varnish-like mass. It is then either weighed as such or dissolved in volumetric acid solution, delivered in measured quantity from a burette, and the excess of the acid titrated with volumetric alkali solution with the use of an indicator. Should the final residual alkaloids still be slightly colored, it is preferable to employ iodeosin as the indicator, as the alkaloidal solution contains ether and the ethereal layer retains

in solution coloring matter or impurity which may be present. If the alkaloids are not colored, hematoxylin or cochineal may safely be used.

The quantity of alkaloid is found by multiplying the number of cubic centimeters of volumetric acid consumed by a constant factor, depending upon the molecular weight of the individual alkaloid. These factors will be found on p. 991, under Tenth-Normal Sulphuric Acid, V.S., and are used throughout the text without explanation. The factor in each case represents the weight in grammes of the alkaloid required to neutralize 1 Cc. of volumetric acid.

OFFICIAL ASSAYS IN WHICH THE ALKALOID IS WEIGHED

ASSAY OF CINCHONA

Recipe.—Cinchona, in No. 80 (or finer) powder, *fifteen grammes*.. 15 Gm.

Ether, sp. gr. not above 0.720 at 25° C. (77° F.),

Chloroform,

Ammonia Water,

Distilled Water,

Normal Sulphuric Acid V.S., each, *a sufficient quantity*.

Introduce the Cinchona into an Erlenmeyer flask or bottle of about 400-Cc. capacity, and add a mixture of 250 Cc. of ether and 50 Cc. of chloroform; then insert the stopper securely, shake the flask vigorously, and allow it to stand for ten minutes. Then add 10 Cc. of ammonia water, and allow it to stand for five hours, shaking at frequent intervals (or continuously with the aid of a mechanical shaker). Next add 15 Cc. of distilled water, shake the flask vigorously, and allow it to stand for a few minutes, so as to cause the powder to settle readily. When the supernatant fluid is quite clear, decant into a measuring flask or cylinder exactly 200 Cc. of the supernatant liquid (representing 10 Gm. of Cinchona), transfer this to a separator and add 15 Cc. of normal sulphuric acid V.S., or sufficient to make the liquid distinctly acid. Shake the separator vigorously for one minute, and allow the two layers of liquid to separate completely. Draw off the lower aqueous layer into a flask. Then add 5 Cc. of normal sulphuric acid V.S. and 5 Cc. of distilled water to the separator and shake it vigorously for about one minute, allow the liquids to separate as before, and again draw off the lower aqueous layer into the flask. Repeat the operation, using 5 Cc. of distilled water in the separator (without acid), drawing off the aqueous liquid into the flask. Filter the combined acid liquids into a measuring cylinder, and wash the filter and flask with enough distilled water to make the contents of the cylinder measure exactly 50 Cc. Pour half (25 Cc.) of the acid liquid into a separator marked No. 1, and the remaining half (25 Cc.) into another separator marked No. 2, which set aside.

I. For Anhydrous Cinchona Alkaloids.—To separator No. 1 (see above) add 25 Cc. of a mixture of chloroform 3 volumes and ether 1 volume, also 5 Cc. of ammonia water, or sufficient to render the liquid alkaline. Insert the stopper and shake the separator carefully for one minute, and then draw off the lower layer into a tared flask or beaker. Add 20 Cc. more of the chloroform-ether mixture to the separator, insert the stopper, and shake the liquid carefully for one minute, again drawing off the lower layer into the tared flask. Repeat the operation with 10 Cc. of chloroform, and draw this off into the tared flask. Evaporate the chloroform-ether solutions in the tared flask or beaker slowly and carefully to dryness on a water-bath. Add 3 Cc. of ether to the dry residue, and again evaporate to dryness. Then place the flask or beaker in an air-bath and heat at 110° C. (230° F.) until the weight after cooling remains constant. This weight in grammes multiplied by 20 will give the percentage of anhydrous cinchona alkaloids (total alkaloids) in the Cinchona.

II. For Ether-soluble Alkaloids.—To separator No. 2 (see above), containing the other 25 Cc. of acid liquid, add 25 Cc. of ether and 5 Cc. of ammonia water, or sufficient to render the liquid alkaline. The temperature of the liquid should be kept below 20° C. (68° F.) by cooling it, if necessary. Shake the separator moderately for two minutes, and allow the liquid to stand for ten minutes at 15° C. (59° F.); after the liquids have separated, draw off and reject the lower aqueous layer and transfer the ethereal liquid to a tared beaker. Add 5 Cc. more of ether to the separator, rinse carefully, and add the rinsings to the tared beaker. Evaporate the ether carefully by the aid of a water-bath, dry the beaker and contents in an air-bath at 110° C. (230° F.) for two hours, cool, and weigh. This weight in grammes multiplied by 20 gives the percentage of the anhydrous ether-soluble alkaloids contained in the Cinchona.

NOTE.—Ether-soluble alkaloids include quinine, quinidine, and cinchonidine.

From the above typical assay it is seen that the process of manipulation is tedious and intricate. It can be noted, however, that there are really three steps in the process: the *extraction* of the drug with ether and chloroform; the *shaking out*, first, with normal sulphuric acid and water, then (after rendering alkaline) with chloroform and ether; and lastly, evaporation of solution and *weighing* of alkaloids. The "separator" mentioned in the assay means one of the separatory funnels pictured on p. 138.

ASSAY OF FLUIDEXTRACT OF CINCHONA

Recipe.—Fluidextract of Cinchona, ten cubic centimeters. . . . 10 Cc.

Ether, sp. gr. not above 0.720 at 25° C. (77° F.),

Normal Sulphuric Acid V.S.,

Ammonia Water,

Chloroform, each, a sufficient quantity.

Transfer 10 Cc. of Fluidextract of Cinchona by means of a graduated pipette to an Erlenmeyer flask of 200-Cc. capacity, and add a mixture of 100 Cc. of ether, 25 Cc. of chloroform, and 10 Cc. of ammonia water. Insert the stopper securely, and shake the flask vigorously, at intervals, during ten minutes. Allow the liquids to separate, decant into a measuring cylinder exactly 66 Cc. of the supernatant liquid (representing 5 Cc. of the Fluidextract), and transfer this to a separator, rinsing the cylinder with 5 Cc. of ether and adding this to the separator. Add to the latter about 10 Cc. of normal sulphuric acid V.S., or enough to make the solution distinctly acid, and shake the separator vigorously for several minutes, and when the liquids have completely separated, draw off the lower layer into a second separator. To the first separator add 5 Cc. more of normal sulphuric acid V.S. and 5 Cc. of distilled water, shake it for several minutes and when the liquids have separated, draw off the lower layer into the second separator. Now add 5 Cc. of distilled water to the first separator, shake it, separate as before, and then draw off the lower aqueous layer into the second separator. To the second separator add 25 Cc. of ether, a small piece of red litmus-paper, and then, gradually, ammonia water, keeping the temperature of the liquids below 25° C. (77° F.) until the reaction is alkaline. Then shake the separator vigorously for two minutes, and allow the liquids to stand for ten minutes at a temperature below 15° C. (59° F.). Draw off and reject the lower aqueous layer, and then transfer the ether-layer into a tared beaker. Add 5 Cc. more of ether to the separator, rinse carefully, and add the rinsings to the tared beaker, and entirely evaporate the ether at a moderate heat on a water-bath. Then dry the beaker in an air-bath at 120° C. (248° F.) for half an hour, cool, and weigh. Replace the beaker in the air-bath and heat again at the same temperature for half an hour, cool, and weigh, repeating until the weight is constant. Multiply the weight of the residue by 20 to obtain the weight in grammes of anhydrous ether-soluble alkaloids contained in one hundred cubic centimeters of the Fluidextract of Cinchona.

ASSAY OF TINCTURE OF CINCHONA.

Transfer 50 Cc. of Tincture of Cinchona to an evaporating dish, and evaporate it on a water-bath until it measures about 10 Cc.; transfer the liquid to a bottle having the capacity of about 180 Cc., rinsing the dish with 10 Cc. of diluted alcohol; then assay the resulting liquid by the method given under *Fluidextractum Cinchonæ* (given above), with the exception that the multiplication of the product should be by 4 instead of 20; the result will represent the weight in grammes of anhydrous ether-soluble alkaloids contained in one hundred cubic centimeters of Tincture of Cinchona.

ASSAY OF COLCHICUM CORM

Recipe.—Colchicum Corm, in No. 60 powder, ten grammes. . . . 10 Gm.

Ether,

Chloroform,

Alcohol,

Ammonia Water,

Distilled Water, each, a sufficient quantity.

Introduce the Colchicum Corm into a 200-Cc. Erlenmeyer flask, and add to it 100 Cc. of a mixture of 77 Cc. of ether, 25 Cc. of chloroform, 8 Cc. of alcohol, and 3 Cc. of ammonia water, insert the stopper securely, and macerate, with frequent shaking, for twelve hours (or preferably for four hours in a mechanical shaker). Filter a sufficient quantity of the liquid into a measuring cylinder until 50 Cc. of filtrate (representing 5 Gm. of Colchicum Corm) have been obtained; then transfer this to a beaker or dish, and evaporate it nearly to dryness by applying a gentle heat. Dissolve the residue in 10 Cc. of ether, add 5 Cc. of water, stir well, and heat gently until the ether has evaporated. After cooling, filter the aqueous solution into a small separator, retaining the insoluble matter as much as possible in the beaker or dish. Redissolve the residue in a little ether, add 5 Cc. of water, and proceed as before. Wash the container and filter with a little water, and shake the combined aqueous solutions well for one minute with 15 Cc. of chloroform. Draw off the chloroform, after perfect separation, into a beaker and again shake out the aqueous liquid successively with three portions of 10 Cc. each of chloroform, collecting these solutions in the beaker. Evaporate the chloroform completely; dissolve the residue in a little alcohol, evaporate the latter, redissolve the residue in 5 Cc. of ether, add 5 Cc. of water, and stir the liquid for a few seconds. Then evaporate the ether on a water-bath containing warm water, and filter the remaining aqueous liquid through a small wetted filter into a separator, washing the dish and filter with 5 Cc. of water, and adding the washings to the separator. Shake out the aqueous liquid with 15 Cc. of chloroform, and when the liquids have separated, draw off the chloroform into a tared flask. Repeat the shaking out successively with three portions of 10 Cc. of chloroform and add each to the tared flask. Evaporate the chloroform completely, dissolve the residue in a little alcohol, evaporate the latter, redissolve the residue in alcohol, evaporate the alcohol as before, and dry the residue at 100° C. (212° F.) until the weight, after cooling, remains constant. The weight of the residue multiplied by 20 gives the percentage of colchicine in the Colchicum Corm.

ASSAY OF COLCHICUM SEED

Recipe.—Colchicum Seed, in No. 60 powder, *ten grammes*. . . . 10 Gm.

Ether,
Chloroform,
Alcohol,
Ammonia Water,
Distilled Water, each, *a sufficient quantity*.

Introduce the Colchicum Seed into a 200-Cc. Erlenmeyer flask, and add to it 100 Cc. of a mixture of 77 Cc. of ether, 25 Cc. of chloroform, 8 Cc. of alcohol, and 3 Cc. of ammonia water, insert the stopper securely, and macerate, with frequent shaking, for twelve hours (or preferably for four hours in a mechanical shaker). Filter a sufficient quantity of the liquid into a measuring cylinder until 50 Cc. of filtrate (representing 5 Gm. of Colchicum Seed) have been obtained; then transfer this to a beaker or dish, and evaporate it nearly to dryness by applying a very gentle heat. Dissolve the residue in 10 Cc. of ether, add 5 Cc. of water, stir well, and heat gently until the ether has evaporated. After cooling, filter the aqueous solution into a small separator, retaining the insoluble matter as much as possible in the beaker or dish. Redissolve the residue in a little ether, add 5 Cc. of water, and proceed as before. Wash the container and filter with a little water, and shake the combined aqueous solutions well for one minute with 15 Cc. of chloroform. Draw off the chloroform, after perfect separation, into a tared flask, and again shake out the aqueous liquid successively with three portions of 10 Cc. each of chloroform, collecting these solutions in the tared flask. Evaporate the chloroform completely; dissolve the residue in a little alcohol; evaporate the latter, redissolve the residue in alcohol, evaporate the alcohol as before, and dry the residue at 100° C. (212° F.) until the weight, after cooling, remains constant. The weight of the residue multiplied by 20 gives the percentage of colchicine in the Colchicum Seed.

ASSAY OF EXTRACT OF COLCHICUM CORM

Recipe.—Extract of Colchicum Corm, *four grammes*. 4 Gm.

Chloroform,
Alcohol,
Ether,
Distilled Water,
Ammonia Water, each, *a sufficient quantity*.

Dissolve the Extract of Colchicum Corm in 20 Cc. of distilled water, carefully transfer the solution to a graduated flask, and add sufficient alcohol to make the liquid measure 100 Cc. Shake the flask well, allow it to stand for five minutes, filter, and collect 50 Cc. of the filtrate (representing 2 Gm. of the Extract), and evaporate it to dryness in a porcelain dish by means of a water-bath. Add to the residue 10 Cc. of ether and 5 Cc. of distilled water, stir the mixture well, and heat gently until the ether is evaporated. After cooling, pour off the aqueous solution, filtering it into a separator, retaining as much of the insoluble matter in the dish as possible. Again treat the residue with 10 Cc. of ether, and 5 Cc. of water, and proceed as before; rinse the dish and filter with a little water and collect all of the aqueous liquids in the separator. Introduce a small piece of red litmus-paper into the separator, add enough ammonia water to render the liquid alkaline, and then shake it out with three successive portions of chloroform, of 20, 15, and 10 Cc. respectively. Collect the combined chloroformic solutions in an Erlenmeyer flask, evaporate the chloroform, and add to the alkaloidal residue two successive small portions of alcohol, evaporating the alcohol each time. Now add to the residue a mixture of 5 Cc. of distilled water and 10 Cc. of ether, agitate the liquid gently and evaporate the ether; after cooling, filter the aqueous liquid into a separator. Rinse the flask with distilled water, pass the rinsings through the filter into the separator, and shake out the aqueous solutions with three successive portions of chloroform, 20, 15, and 10 Cc. respectively. Collect the combined chloroformic solutions in a tared Erlenmeyer flask, evaporate the chloroform, and treat the alkaloidal residue with two successive small portions of alcohol, evaporating the alcohol each time, and dry the residue, at 100° C. (212° F.), to a constant weight. The weight multiplied by 50 will give the percentage of colchicine in the Extract of Colchicum Corm.

ASSAY OF FLUIDEXTRACT OF COLCHICUM SEED

Recipe.—Fluidextract of Colchicum Seed, *ten cubic centimeters*. 10 Cc.

Ether,
Chloroform,
Alcohol,
Ammonia Water,
Distilled Water, each, *a sufficient quantity*.

Measure into a separator 10 Cc. of Fluidextract of Colchicum Seed, add 1 Cc. of ammonia water, and shake out the alkaloid with three successive portions, 15, 15, and 10 Cc., of chloroform. Collect the chloroformic solutions in a beaker or dish, and evaporate it nearly to dryness by applying a very gentle heat. Dissolve the residue in 10 Cc. of ether, add 5 Cc. of water, stir well, and heat gently until the ether is evaporated. After cooling, filter the aqueous solution into a small separator, retaining the insoluble matter as much as possible in the beaker or dish. Redissolve the residue in a little ether, add 5 Cc. of water, and proceed as before. Wash the container and filter with a little water, and shake the aqueous solution well for one minute with 15 Cc. of chloroform. Draw off the chloroform, after perfect separation, into a tared flask, and again shake out the aqueous liquid, successively, with three portions of 10 Cc. each of chloroform, collecting these solutions in the tared flask. Evaporate the chloroform completely; dissolve the residue in a little alcohol, evaporate the latter, redissolve it in alcohol, evaporate the alcohol as before, and dry the residue at 100° C. (212° F.) until the weight, after cooling in a desiccator, remains constant. Multiply the weight of the residue by 10 to obtain the weight in grammes of colchicine contained in *one hundred cubic centimeters* of the Fluidextract of Colchicum Seed.

ASSAY OF TINCTURE OF COLCHICUM SEED

Transfer 100 Cc. of Tincture of Colchicum Seed to an evaporating dish, and evaporate it on a water-bath until it measures about 10 Cc. Add, if necessary, sufficient alcohol to dissolve any separated substance, and then assay the resulting liquid by the method given under *Fluidextractum Colchici Seminis* (just above), with the exception that the multiplication of the product by 10 be omitted; the result will represent the weight in grammes of colchicine contained in *one hundred cubic centimeters* of Tincture of Colchicum Seed.

ASSAY OF CONIUM

Recipe.—Conium, in No. 60 powder, *ten grammes*..... 10 Gm.
 Ether,
 Alcohol,
 Ammonia Water,
 Normal Sulphuric Acid V.S.,
 Sodium Carbonate T.S.,
 Distilled Water,
 Hydrochloric Acid Solution (5 per cent. HCl), each,
a sufficient quantity.

Place the Conium in a 200-Cc. Erlenmeyer flask, add 100 Cc. of a mixture of ether 98 parts, alcohol 8 parts, and ammonia water 3 parts (by volume), insert the stopper securely, and shake the flask at intervals during four hours. After the powder has settled, decant 50 Cc. of the clear liquid into a beaker (representing 5 Gm. of Conium), and add sufficient normal sulphuric acid V.S. to produce a distinctly acid reaction. Evaporate the ether at a gentle heat by the aid of a water-bath; then add 15 Cc. of alcohol, and set the beaker aside in a cool place for two hours to allow the ammonium sulphate to deposit. Filter the liquid; wash the residue and filter with a little alcohol, and add the washings to the filtrate; neutralize any excessive amount of acid with sodium carbonate T.S., being careful to retain a slight acidity. Concentrate the liquid to 3 Cc. by the aid of a gentle heat on a water-bath, add 3 Cc. of distilled water and 2 drops of normal sulphuric acid V.S. Add 15 Cc. of ether to remove traces of fatty matter, pour off the ether solution, and repeat the washings with 15 Cc. of ether. Then transfer the acid liquid to a separator, introduce a small piece of red litmus-paper, and add sufficient sodium carbonate T.S. to render the liquid slightly alkaline; then shake out with successive portions of 15, 15, and 10 Cc. of ether. To the combined ether solutions, in a tared beaker, add, drop by drop, sufficient hydrochloric acid solution (5 per cent.) to insure an excess of acid, and then evaporate the ether by a gentle heat on a water-bath. Remove the excess of hydrochloric acid by adding to the residue 3 Cc. of alcohol and heating gently to evaporate the liquid, repeat this operation once, and dry the residue at a temperature not exceeding 60° C. (140° F.) until the weight, after cooling in a desiccator, remains constant. The weight of the residue multiplied by 0.777, and this product by 20, gives the percentage of coniine contained in the Conium.

ASSAY OF FLUIDEXTRACT OF CONIUM

Recipe.—Fluidextract of Conium, *ten cubic centimeters*..... 10 Cc.
 Ether,
 Alcohol,
 Absolute Alcohol,
 Ammonia Water,
 Normal Sulphuric Acid V.S.
 Sodium Carbonate T.S.,
 Hydrochloric Acid Solution (5 per cent. HCl),
 Distilled Water, each, *a sufficient quantity.*

Transfer 10 Cc. of Fluidextract of Conium by means of a graduated pipette to an evaporating dish containing a little clean sand, and evaporate it to dryness at a gentle heat. Mix the sand uniformly with the extract and transfer it to an Erlenmeyer flask of about 200-Cc. capacity, rinse the dish with 100 Cc. of a mixture of ether 100 Cc., alcohol 7 Cc., and ammonia water 3 Cc., added in portions, and transfer the rinsings to the flask. Insert the stopper securely and shake the flask at intervals during one hour. Decant 50 Cc. of the liquid (representing 5 Cc. of the Fluidextract of Conium) into a beaker, and add sufficient normal sulphuric acid V.S. to produce a distinctly acid reaction. Evaporate the ether at a gentle heat by the aid of a water-bath; then add 15 Cc. of absolute alcohol, and set the beaker aside in a cool place for two hours to allow the ammonium sulphate to deposit. Filter the liquid; wash the residue and filter with a little absolute alcohol, and add the washing to the filtrate, neutralize any excessive amount of acid with sodium carbonate T.S., being careful to retain a slight acidity. Concentrate the liquid to 3 Cc. by the aid of a gentle heat on a water-bath, add 3 Cc. of distilled water and 2 drops of normal sulphuric acid V.S. Add 15 Cc. of ether to remove traces of fatty matter, pour off

the ether solution, and repeat the washing. Then transfer the acid liquid to a separator, introduce a small piece of red litmus-paper, and add sufficient sodium carbonate T.S. to render the liquid slightly alkaline; then shake out with successive portions of 15, 10, and 10 Cc. of ether. To the combined ether solutions in a tared beaker add, drop by drop, sufficient hydrochloric acid solution (5 per cent.) to insure an excess of acid, and then evaporate the ether by a gentle heat on a water-bath. Remove the excess of hydrochloric acid by adding to the residue 3 Cc. of alcohol and heating gently to evaporate the liquid, repeat this operation once, and dry the residue at a temperature not exceeding 60° C. (140° F.) until the weight, after cooling in a desiccator, remains constant. Multiply the weight of the residue by 0.777, and the product by 20, to obtain the weight in grammes of coniine contained in *one hundred cubic centimeters* of the Fluidextract of Conium.

ASSAY OF GUARANA

Recipe.—Guarana, in No. 60 powder, *six grammes*. 6 Gm.
Chloroform,
Ether,
Ammonia Water,
Normal Sulphuric Acid V.S.,
Distilled Water, each, *a sufficient quantity*.

Introduce the Guarana into an Erlenmeyer flask, and pour upon it 120 Cc. of chloroform and 6 Cc. of ammonia water, and insert the stopper securely. Shake the flask at intervals for half an hour, and allow it to stand for four hours. Filter a sufficient quantity of the liquid into a measuring cylinder until 100 Cc. (representing 5 Gm. of Guarana) have been obtained, then transfer the filtrate to a flask, and distil off all the chloroform by means of a water-bath. Dissolve the alkaloidal residue in a mixture of 2 Cc. of normal sulphuric acid V.S. and 20 Cc. of warm distilled water. Allow the liquid to cool, and filter it into a separator, rinse the flask and filter with several small portions of distilled water, add 20 Cc. of chloroform and 2 Cc. of ammonia water to the separator, and shake it for one minute. Draw off the chloroform into a tared flask and repeat the extraction with two portions of 10 Cc. each of chloroform. Distil off the chloroform from the combined liquids, and when the residue is dry add 2 Cc. of ether and evaporate on a water-bath very carefully to avoid decrepitation; continue the heating until the weight of the residue after cooling remains constant. This weight multiplied by 20 will give the percentage of the alkaloidal principles contained in the Guarana.

ASSAY OF FLUIDEXTRACT OF GUARANA

Recipe.—Fluidextract of Guarana, *five cubic centimeters*. 5 Cc.
Chloroform,
Ether,
Ammonia Water,
Normal Sulphuric Acid V.S.,
Distilled Water, each, *a sufficient quantity*.

Transfer to a separator 5 Cc. of Fluidextract of Guarana, add 15 Cc. of chloroform and 1 Cc. of ammonia water. Shake well and allow the liquid to separate completely. Draw off the chloroform into a beaker. Shake out the fluid remaining in the separator with two additional portions of chloroform of 10 Cc. each, evaporate the combined chloroformic solutions carefully to dryness. Dissolve the alkaloidal residue in a mixture of 2 Cc. of normal sulphuric acid V.S. and 20 Cc. of warm distilled water. Allow it to cool, and filter the solution into a separator, rinse the flask and filter with distilled water, adding the rinsings to the separator, then add 20 Cc. of chloroform and 2 Cc. of ammonia water and shake the separator for one minute. Draw off the chloroform into a tared flask, and repeat the extraction with two portions of 10 Cc. each of chloroform, adding this to the tared flask. Distil off the chloroform, and, when dry, add 2 Cc. of ether, and evaporate this very carefully with the aid of a water-bath (to avoid decrepitation). Dry the residue to a constant weight on the water-bath. Multiply the weight by 20, which will give the weight in grammes of alkaloids contained in *one hundred cubic centimeters* of Fluidextract of Guarana.

ASSAY OF HYDRASTIS

Recipe.—Hydrastis, in No. 60 powder, fifteen grammes. 15 Gm.
 Ether,
 Ammonia Water,
 Distilled Water,
 Normal Sulphuric Acid V.S. each, a sufficient quantity.

Introduce the Hydrastis into an Erlenmeyer flask of 250-Cc. capacity, add 150 Cc. of ether, shake the flask during ten minutes, and add 5 Cc. of ammonia water, again shaking the flask at intervals during half an hour. Then add 15 Cc. of distilled water to the mixture in the flask and shake it until the drug collects in masses, and at once pour off, into a measuring cylinder, 100 Cc. of the supernatant ether solution and transfer it to a separator. Add 15 Cc. of normal sulphuric acid V.S. to the separator, and shake it moderately during one minute. Allow the liquids to separate, and draw off the lower acid liquid into a second separator. Again shake out the ether solution with 5 Cc. of normal sulphuric acid V.S. and 5 Cc. of distilled water, and shake the separator for one minute. After the liquids have separated, draw off the acid solution as before into the second separator. Repeat the same process with 5 Cc. of distilled water, drawing this also into the second separator. Introduce a small piece of red litmus-paper into the second separator, add enough ammonia water to render the liquid alkaline, and then 25 Cc. of ether, and shake the separator moderately during one minute, and when the liquids have separated draw off the lower alkaline liquid into another separator, and the ether solution into a tared beaker. Again shake out the alkaline liquid, using 20 Cc. of ether, shake the separator for one minute, and when the liquids have separated, draw off the alkaline liquid into the other separator, and the ether solution into the tared beaker. Finally, again shake out the alkaline liquid, using 15 Cc. of ether, proceeding as before, and adding the ether solution to the liquid in the tared beaker. Evaporate the ether carefully with the aid of a water-bath, and dry the alkaloidal residue in the beaker to a constant weight at 100° C. (212° F.). The weight found, multiplied by 10, will give the percentage of hydrastine in the Hydrastis.

ASSAY OF FLUIDEXTRACT OF HYDRASTIS

Recipe.—Fluidextract of Hydrastis, ten cubic centimeters. . . . 10 Cc.
 Distilled Water,
 Potassium Iodide,
 Ammonia Water,
 Ether, each, a sufficient quantity.

Transfer 10 Cc. of Fluidextract of Hydrastis by means of a graduated pipette to a 100-Cc. measuring flask, add 85 Cc. of distilled water in which 2 Gm. of potassium iodide have been previously dissolved, and sufficient water to make 100 Cc., and shake the liquid for several minutes. Then filter, 50 Cc. of the liquid into a measuring cylinder and transfer it to a separator. Render the liquid alkaline with ammonia water, add 30 Cc. of ether, and shake the separator at intervals during several minutes. When separated, draw off the aqueous layer into a beaker and the ether solution into a tared beaker. Return the aqueous solution to the separator, and shake it with 20 Cc. more of ether for one minute. Draw off and reject the aqueous layer, and run the ether solution into the tared beaker. Allow the combined ether solutions to evaporate at a gentle heat, and dry the residue in the beaker to a constant weight on a water-bath. Multiply the weight by 20, which will give the weight in grammes of hydrastine contained in *one hundred cubic centimeters* of Fluidextract of Hydrastis.

ASSAY OF TINCTURE OF HYDRASTIS

Transfer 100 Cc. of Tincture of Hydrastis to an evaporating dish, and evaporate it on a water-bath until the liquid measures about 10 Cc. If any insoluble matter has separated, add sufficient alcohol to dissolve it, and then assay the resulting liquid by the method given under *Fluidextractum Hydrastis* (see just above), using the same details as there directed for 10 Cc. of Fluidextract of Hydrastis, with the exception that the weight of the residual alkaloids must be multiplied by 2 instead

of by 20, as there directed, to give the weight in grammes of hydrastine contained in one hundred cubic centimeters of Tincture of Hydrastis.

ASSAY OF OPIUM

Recipe.—Opium, in any condition to be valued, *ten grammes.* 10.0 Gm.
Ammonia Water, three and one-half cubic centimeters, 3.5 Cc.
Alcohol,
Ether,
Distilled Water,
Lime Water, each, *a sufficient quantity.*

Introduce the Opium (which, if fresh, should be in very small pieces, and if dry, in very fine powder) into an Erlenmeyer flask having a capacity of about 300 Cc., add 100 Cc. of distilled water, stopper the flask, and agitate it every ten minutes (or continuously in a mechanical shaker) during three hours. Then pour the contents as evenly as possible upon a wetted filter having a diameter of 12 Cm., and, when the liquid has drained off, wash the residue with distilled water, carefully dropped upon the edges of the filter and its contents, until 150 Cc. of filtrate have been obtained. Then carefully transfer the moist Opium back to the flask by means of a spatula, add 50 Cc. of distilled water, agitate it thoroughly and repeatedly during fifteen minutes, and return the whole to the filter. When the liquid has drained off, wash the residue, as before, until the second filtrate measures 150 Cc., and finally collect about 20 Cc. more of a third filtrate. Evaporate carefully in a tared dish, first, the second filtrate to a small volume, then add the first filtrate, rinsing the vessels with the third filtrate, and continue the evaporation until the residue weighs 14 Gm. Rotate the concentrated solution about in the dish until the rings of extract are redissolved, pour the liquid into a tared Erlenmeyer flask having a capacity of about 100 Cc., and after the rinsings have been added to the flask, weighs 20 Gm. Then add 10 Gm. (or 12.2 Cc.) of alcohol, shake the flask well, add 25 Cc. of ether, and repeat the shaking. Now add the ammonia water from a graduated pipette or burette, stopper the flask with a sound cork, shake it thoroughly during ten minutes, and then set it aside, in a moderately cool place, for at least sixteen hours.

Remove the stopper carefully, and should any crystals adhere to it, brush them into the flask. Place in a small funnel two rapidly acting filters, of a diameter of 7 Cm., plainly folded, one within the other (the triple fold of the inner filter being laid against the single side of the outer filter), wet them well with ether, and decant the ethereal solution as completely as possible upon the inner filter. Add 10 Cc. of ether to the contents of the flask, rotate it, and again decant the ethereal layer upon the inner filter. Repeat this operation with another portion of 10 Cc. of ether. Then pour the liquid in the flask into the filter, in portions, in such a way as to transfer the greater portion of the crystals to the filter, and, when the liquid has passed through, transfer the remaining crystals to the filter by washing the flask with several portions of water, using not more than 15 Cc. in all. Use a feather or rubber-tipped glass rod to remove the crystals that adhere to the flask. Allow the double filter to drain, then apply water to the crystals, drop by drop, until they are practically free from mother-liquor, and afterward wash them, drop by drop, from a pipette, with alcohol previously saturated with powdered morphine. When this has passed through, displace the remaining alcohol by ether, using about 10 Cc. or more, if necessary. Allow the filter to dry in a moderately warm place, at a temperature not exceeding 60° C. (140° F.), until its weight remains constant, then carefully transfer the crystals to a tared watch-glass and weigh them.

Place the crystals (which are not quite pure) in an Erlenmeyer flask, add lime water (10 Cc. for each 0.1 Gm. of morphine) and shake the flask at intervals during, half an hour. Pass the liquid through two counterpoised rapidly acting, plainly folded filters, one within the other (the triple fold of the inner filter being laid against the single fold of the outer filter), rinse the flask with more lime water and pass the washings through the filter until the filtrate, after acidulating, no longer yields a precipitate with mercuric potassium iodide T.S. Press the filters until nearly dry between bibulous paper and dry them to a constant weight, then weigh the contents, using the outer filter as a counterpoise. Deduct the weight of the insoluble matter on the filter from the weight of the impure morphine previously found. The difference, multiplied by 10, represents the percentage of crystallized morphine contained in the Opium.

ASSAY OF EXTRACT OF OPIUM

Recipe.—**Extract of Opium**, dried at 100° C. (212° F.), *four grammes*..... **4.0 Gm.**
Ammonia Water, two and two-tenths cubic centimeters.. **2.2 Cc.**
Alcohol,
Ether,
Water,
Lime Water, each, a sufficient quantity.

Dissolve the Extract of Opium in 30 Cc. of water, filter the solution through a small filter, and wash the filter and residue with water, until all soluble matters are extracted, collecting the washings separately. Evaporate, in a tared dish, on a water-bath, first, the washings to a small volume, then add the first filtrate, and evaporate the whole to a weight of 10 Gm. Rotate the concentrated solution in the dish until the rings of extract are redissolved, pour the liquid into a tared Erlenmeyer flask having a capacity of about 100 Cc., and rinse the dish with a few drops of water at a time, until the entire solution weighs 15 Gm. Add 7 Gm. (or 8.5 Cc.) of alcohol, shake the flask well, then add 20 Cc. of ether, and repeat the shaking. Now add the ammonia water from a graduated pipette or burette, stopper the flask with a sound cork, shake it thoroughly during ten minutes, and then set it aside, in a moderately cool place, for at least six hours or over night.

Remove the stopper carefully, and, should any crystals adhere to it, brush them into the flask. Place in a small funnel two rapidly acting filters, of a diameter of 7 Cm., plainly folded, one within the other (the triple fold of the inner filter being laid against the single side of the outer filter), wet them well with ether, and decant the ether solution as completely as possible upon the inner filter. Add 15 Cc. of ether to the contents of the flask, rotate it, and again decant the ethereal layer upon the inner filter. Repeat this operation with another portion of 15 Cc. of ether. Then pour into the filter the liquid in the flask, in portions, in such a way as to transfer the greater portion of the crystals to the filter, and, when the liquid has passed through, transfer the remaining crystals to the filter by washing the flask with several portions of water, using not more than about 10 Cc. in all. Allow the double filter to drain, then apply water to the crystals, drop by drop, until they are practically free from mother-liquor, and afterward wash them with alcohol (previously saturated with powdered morphine), added drop by drop from a pipette. When this has passed through, displace the remaining alcohol by ether, using about 10 Cc. or more if necessary. Allow the filter to dry in a moderately warm place, at a temperature not exceeding 60° C. (140° F.), until its weight remains constant, then carefully transfer the crystals of morphine to a tared watch-glass and weigh them.

Transfer the crystals (which are not quite pure) to an Erlenmeyer flask, add lime water in the proportion of 10 Cc. for each 0.1 Gm. of morphine, and shake the flask at intervals for twenty-five minutes. Pass the liquid through two counterpoised rapidly acting, plainly folded filters, one within the other (the triple fold of the inner filter being laid against the single side of the outer filter), and rinse the flask with additional lime water, passing the washings through the filter until the filtrate, after acidulation, no longer yields a precipitate with mercuric potassium iodide T.S. Then press the filters between bibulous paper, dry them to a constant weight, and weigh the contents of the inner filter, using the outer as a counterpoise. Subtract the weight of the insoluble matter on the filter from the weight of the impure morphine previously found, and the difference, multiplied by 25, will be the percentage of pure crystalline morphine contained in the Extract of Opium.

ASSAY OF TINCTURE OF OPIUM

Recipe.—**Tincture of Opium**, one hundred cubic centimeters. **100.0 Cc.**
Ammonia Water, three and one-half cubic centimeters, **3.5 Cc.**
Alcohol,
Ether,
Water,
Lime Water, each, a sufficient quantity.

Transfer 100 Cc. of Tincture of Opium to an evaporating dish and evaporate it on a water-bath to about 20 Cc., add 40 Cc. of water, mix thoroughly, and set the liquid aside for one hour, occasionally stirring to disintegrate the resinous flakes

adhering to the dish. Then filter the liquid and wash the filter and residue with water, until all soluble matter is extracted (indicated by an almost colorless filtrate), and collect the washings separately. First evaporate the washings, in a tared dish, to a small volume, then add the first filtrate and evaporate the whole to a weight of 14 Gm.

Determine the morphine in this extract by the method given under Opium (p. 1025 twenty-first line of the Assay, beginning with the word "Rotate"), using the same details as there directed for 10 Gm. of Opium, with the exception that the final multiplication by 10 be omitted. The result will represent the weight in grammes of crystallized morphine yielded by *one hundred cubic centimeters* of Tincture of Opium.

ASSAY OF TINCTURE OF DEODORIZED OPIUM

If 100 Cc. of Tincture of Deodorized Opium be assayed by the process given under *Tinctura Opii* (p. 1027), it should yield not less than 1.2 nor more than 1.25 Gm. of crystallized morphine.

These assays are reproduced exactly as given in the pharmacopœia because condensation is difficult and tabulation almost impossible.

Jalap is assayed by extraction and weighing, but as the assay is a purely physical process, involving no chemical change and no "shaking out," it is placed under the discussion of the drug on p. 717.

It might be well to here explain the seeming discrepancy in the statements on pp. 717 and 718 relating to the resin of jalap. On p. 717 we learn that jalap should contain 7 per cent. of resin, of which not more than 15 per cent. should be soluble in ether. This clearly means that 15 per cent. of 7 per cent. or 1.05 per cent. of the weight of the drug should be resin soluble in ether.

OFFICIAL ASSAYS IN WHICH THE ALKALOID IS TITRATED

ASSAY OF ACONITE

Recipe.—Aconite, in No. 40 powder, *ten grammes*. 10 Gm.

Alcohol,
Distilled Water,
Ether,
Ammonia Water,
Tenth-normal sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S., each, *a sufficient quantity*.

Introduce the Aconite into a 200-Cc. Erlenmeyer flask, add 75 Cc. of a mixture of alcohol, 7 parts, and distilled water, 3 parts (by volume), stopper the flask securely, and agitate it at intervals during four hours. After placing a pledget of cotton in the bottom of a small cylindrical glass percolator (25 Mm. in diameter), carefully transfer the contents of the flask to the percolator. When the liquid has all passed through, continue the percolation with more of the same mixture until 150 Cc. of percolate have been obtained. Pour the percolate into a shallow porcelain evaporating dish, and evaporate it to dryness at a temperature not exceeding 60° C. (140° F.). Add 5 Cc. of tenth-normal sulphuric acid V.S. and 25 Cc. of distilled water. When the extract is dissolved, filter the liquid into a separator, washing the dish and filter with about 25 Cc. of distilled water, and add the washings to the separator. Add 25 Cc. of ether and 2 Cc. of ammonia water to the separator, and agitate it for one minute. Draw off the lower layer into a flask and filter the ether solution into a beaker. Return the contents of the flask to the separator, add 15 Cc. of ether, and again agitate it for one minute. Draw off the lower layer into the flask and filter the ether solution into the beaker. Repeat the shaking out with two other portions of 10 Cc. each of ether. Evaporate the combined ether solutions to dryness and dissolve the residue in 3 Cc. of tenth-normal sulphuric acid V.S. Add to the solution 5 drops of cochineal T.S., and then carefully run in fiftieth-normal potassium hydroxide

V.S. until a pink color is produced. Divide the number of Cc. of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 3 (the 3 Cc. of tenth-normal sulphuric acid V.S. taken), multiply the remainder by 0.064, and this product by 10, which will give the percentage of aconitine in the Aconite.

ASSAY OF FLUIDEXTRACT OF ACONITE

Recipe.—Fluidextract of Aconite, *ten cubic centimeters*. 10 Cc.

Ether,
Distilled Water,
Ammonia Water,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S., each, *a sufficient quantity*.

Transfer 10 Cc. of Fluidextract of Aconite by means of a graduated pipette to a porcelain dish, and evaporate it carefully to dryness on a water-bath at a temperature not exceeding 60° C. (140° F.). Add 5 Cc. of tenth-normal sulphuric acid V.S. and 25 Cc. of distilled water. When the extract is dissolved, filter the liquid into a separator, washing the dish and filter with about 25 Cc. of distilled water; when this has passed through, add 25 Cc. of ether and 2 Cc. of ammonia water to the separator, and agitate for one minute. Draw off the lower layer into a flask and filter the ether solution into a beaker. Return the contents of the flask to the separator, add 15 Cc. of ether, and agitate for one minute. Draw off the lower layer into the flask and filter the ether solution into the beaker. Repeat, with two other portions of 10 Cc. each of ether. Evaporate the ether solution to dryness and dissolve the residue in 3 Cc. of tenth-normal sulphuric acid V.S. diluted with 20 Cc. of distilled water. Add to the solution 5 drops of cochineal T.S., and then carefully run in fiftieth-normal potassium hydroxide V.S. until a pink color is produced. Divide the number of Cc. of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 3 (the 3 Cc. of tenth-normal sulphuric acid V.S. taken), multiply the remainder by 0.064, and this product by 10, which will give the weight in grammes of aconitine contained in *one hundred cubic centimeters* of the Fluidextract of Aconite.

ASSAY OF TINCTURE OF ACONITE

Transfer 100 Cc. of Tincture of Aconite to an evaporating dish and evaporate it carefully to dryness at a temperature not exceeding 60° C. (140° F.), and assay the resulting extract by the method given under *Fluidextractum Aconiti* (above), use the same details as there directed for 10 Cc. of Fluidextract of Aconite, with the exception that the multiplication of the product by 10 must be omitted; the result will represent the weight in grammes of aconitine contained in *one hundred cubic centimeters* of Tincture of Aconite.

ASSAY OF BELLADONNA LEAVES

Recipe.—Belladonna Leaves, in No. 60 powder, *ten grammes*. . . 10 Gm.

Chloroform,
Ether,
Normal Sulphuric Acid V.S.,
Ammonia Water,
Distilled Water,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S. or Iodeosin T.S., each, *a sufficient quantity*.

Place the Belladonna Leaves in an Erlenmeyer flask, and add 50 Cc. of a mixture of chloroform 1 part and ether 4 parts (both by volume). After inserting the stopper securely, allow the flask to stand ten minutes, then add 2 Cc. of ammonia water mixed with 3 Cc. of distilled water, and shake the flask well at frequent intervals during one hour. Then transfer as much as possible of the contents of the flask to a small percolator which has been provided with a pledget of cotton packed firmly in the neck and inserted in a separator containing 6 Cc. of normal sulphuric

acid V.S. diluted with 20 Cc. of distilled water. When the liquid has passed through the cotton, pack the Belladonna Leaves firmly in the percolator with the aid of a glass rod, and having rinsed the flask with 10 Cc. of the chloroform-ether mixture, transfer the remaining contents of the flask to the percolator, by the aid of several small portions (5 Cc.) of the chloroform-ether mixture, and continue the percolation with successive small portions of the same liquid (using in all 50 Cc.). Next, shake the separator well for one minute, after securely inserting the stopper, and when the liquids have completely separated, draw off the acid solution into another separator. Add to the chloroform-ether mixture 10 Cc. of sulphuric acid mixture of the same strength as that previously used, agitate well, and again draw off the acid solution into the second separator; repeat this operation once more, drawing off the acid solution as before; introduce into the acid solutions contained in the second separator a small piece of red litmus-paper, then add ammonia water until the liquid is distinctly alkaline, and shake out with three successive portions of chloroform, 15, 15, and 5, Cc.; collect the chloroform solutions in a beaker, place it on a water-bath containing warm water, and allow the chloroform to entirely evaporate. Dissolve the residue in 3 Cc. of ether, and let this also evaporate completely. To the alkaloidal residue add 3 Cc. of tenth-normal sulphuric acid V.S. and 5 drops of cochineal T.S. (or iodeosin T.S.), then titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 3 (the 3 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0287, and this product by 10; the result will be the percentage of total mydriatic alkaloids contained in the Belladonna Leaves.

ASSAY OF BELLADONNA PLASTER (RUBBER BASE)

Recipe.—Belladonna Plaster, spread upon cloth, *ten grammes*, 10 Gm.

Chloroform,
Ammonia Water,
Alcohol,
Distilled Water,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S., each, a *sufficient quantity*.

Into a suitable beaker containing 50 Cc. of chloroform and 3 Cc. of ammonia water, introduce the Belladonna Plaster cut into strips. Stir until the Plaster is entirely removed from the cloth; then pour off the chloroform into another beaker, wash the cloth with 25 Cc. of chloroform and 1 Cc. of ammonia water carefully, and add the washings to the chloroformic solution first obtained. If necessary, repeat the washing with 25 Cc. of chloroform and add this also to the chloroformic solution. Then dry the cloth at a low temperature; cool and weigh it, and subtract its weight from the original weight of the Plaster. To the chloroformic solution add four-fifths of its volume of alcohol, stir gently, and allow the liquid to stand until all of the rubber has separated in a compact mass. Then pour off the supernatant liquid into a separator of 250-Cc. capacity, and, having prepared a solution of sulphuric acid by diluting 40 Cc. of normal sulphuric acid V.S. with 60 Cc. of distilled water, add 20 Cc. of the solution to the separator, and agitate for two minutes, rotating gently. Draw off the chloroformic solution into another separator, shake this with 10 Cc. of the sulphuric acid solution, and add the acid solution to that in the first separator. Repeat until the acid washings cease to give a reaction with mercuric potassium iodide T.S.; combine the acid liquids, and, having rendered this solution alkaline with ammonia water, shake out the alkaloids with three successive portions of 25, 15, and 10 Cc. of chloroform. Collect these in a flask, distil off all of the chloroform with the aid of a water-bath. To the alkaloidal residue add a slight excess of tenth-normal sulphuric acid V.S., noting the quantity used, and then add 10 drops of chloroform and, after rotating, evaporate the latter by means of a water-bath. Then add 5 drops of cochineal T.S., and, rotating, titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from the number of cubic centimeters of tenth-normal sulphuric acid V.S. first added, and divide the difference by the number of grammes of Belladonna Plaster separated

from the cloth; multiply the quotient by 0.0287, and this product by 100, which will give the percentage of mydriatic alkaloids in the Belladonna Plaster.

ASSAY OF EXTRACT OF BELLADONNA LEAVES

Recipe.—Extract of Belladonna Leaves, five grammes..... 5 Gm.

Alcohol,
Ammonia Water,
Distilled Water,
Chloroform,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S. or Iodeosin T.S., each, a sufficient quantity.

Introduce the Extract of Belladonna Leaves into a small beaker and dissolve it in a mixture consisting of alcohol 5 Cc., distilled water 10 Cc., ammonia water 2 Cc., and chloroform 20 Cc. When dissolved, transfer it to a separator, rinsing the beaker with a little alcohol and adding the rinsings to the separator. Insert the stopper securely and shake the separator for half a minute. Draw off the chloroformic layer into a second separator, and add to the first separator 10 Cc. more of chloroform. Shake it for half a minute, allow to separate, and again draw off the chloroformic layer into the second separator. Repeat this with 10 Cc. more of chloroform. To the united chloroformic liquids in the second separator, add 5 Cc. of normal sulphuric acid V.S. and 10 Cc. of distilled water, and shake it for half a minute. Draw off the chloroformic layer, after the liquids have separated, into the first separator, after cleaning it thoroughly, and the aqueous layer into a beaker, and repeat the process by adding to the first separator 10 Cc. of distilled water and 1 Cc. of normal sulphuric acid V.S. Draw off the chloroformic layer, rejecting the same, and then run the acid aqueous layer into the beaker. Pass the combined acid aqueous solutions through a pledget of purified cotton into the first separator, after cleaning it thoroughly, rinsing the second separator, the beaker, and the funnel with about 10 Cc. of distilled water. To the first separator, add 15 Cc. of chloroform, a small piece of red litmus-paper, and enough ammonia water to produce a distinctly alkaline reaction. Shake the separator for half a minute, and when the liquids have separated, draw off the chloroformic layer into a beaker. Repeat this process with two portions of 10 Cc. each of chloroform, and evaporate the combined chloroformic liquids in the beaker to dryness on a water-bath containing warm water; dissolve the residue in 3 Cc. of ether and allow the latter to evaporate completely. To the alkaloidal residue add 5 Cc. of tenth-normal sulphuric acid V.S. and 5 drops of cochineal T.S. (or iodeosin T.S.), then titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 5 (the 5 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0287, and this product by 20, to obtain the percentage of mydriatic alkaloids contained in the Extract of Belladonna Leaves. The figure 0.0287 represents the weight in grammes of mydriatic alkaloids (mainly atropine) required to neutralize 1 Cc. of tenth-normal sulphuric acid V.S.

ASSAY OF TINCTURE OF BELLADONNA LEAVES

Transfer 100 Cc. of Tincture of Belladonna Leaves to an evaporating dish and evaporate it on a water-bath until it measures about 10 Cc. Add, if necessary, sufficient alcohol to dissolve any separated substance, and then assay the resulting liquid by the method given under *Fluidextractum Belladonnæ Radicis* (p. 1031), using the same details as there directed for 10 Cc. of Fluidextract of Belladonna Root, with the exception that the multiplication of the product by 10 be omitted; the result will represent the weight in grammes of alkaloids contained in one hundred cubic centimeters of Tincture of Belladonna Leaves.

ASSAY OF BELLADONNA ROOT

The method to be employed is identical with that given on p. 1028 for Belladonna Leaves, using *ten grammes* of Belladonna Root in No. 60 powder.

ASSAY OF FLUIDEXTRACT OF BELLADONNA ROOT

Recipe.—Fluidextract of Belladonna Root, *ten cubic centimeters*..... 10 Cc.
 Distilled Water,
 Ammonia Water,
 Chloroform,
 Normal Sulphuric Acid V.S.,
 Tenth-normal Sulphuric Acid V.S.,
 Fiftieth-normal Potassium Hydroxide V.S.,
 Cochineal T.S. or Iodeosin T.S., each, *a sufficient quantity*.

Transfer 10 Cc. of Fluidextract of Belladonna Root by means of a graduated pipette to a separator, add 10 Cc. of distilled water, 20 Cc. of chloroform, and 2 Cc. of ammonia water. Shake the separator well for one minute, and draw off the lower chloroformic layer into a second separator. Repeat the extraction with two portions of 10 Cc. each of chloroform, and draw the chloroformic solution into the second separator. To the latter add 8 Cc. of normal sulphuric acid V.S. and 20 Cc. of distilled water, shaking well for one minute. When perfectly separated, draw off and reject the lower chloroformic layer, and filter the acid aqueous layer into a clean separator. Wash the separator and filter with 10 Cc. of distilled water, adding this to the clean separator. To the latter add 20 Cc. of chloroform and 4 Cc. of ammonia water, and shake well for several minutes. Draw off the lower chloroformic layer into a beaker and repeat the extraction with two portions of 10 Cc. each of chloroform, adding the chloroformic solution to the beaker. Allow the chloroform in the beaker to evaporate on a water-bath, containing warm water, until the residue is perfectly dry. To the alkaloidal residue add 5 Cc. of tenth-normal sulphuric acid V.S., and when the residual alkaloids have all dissolved, titrate the solution with fiftieth-normal potassium hydroxide V.S., using 5 drops of cochineal or iodeosin T.S. as an indicator. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 5 (the 5 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0287, and this product by 10, to obtain the weight in grammes of mydriatic alkaloids contained in *one hundred cubic centimeters* of the Fluidextract of Belladonna Root.

ASSAY OF COCA

Recipe.—Coca, in No. 60 powder, *ten grammes*..... 10 Gm.
 Chloroform,
 Ether,
 Normal Sulphuric Acid V.S.,
 Ammonia Water,
 Distilled Water,
 Tenth-normal Sulphuric Acid V.S.,
 Fiftieth-normal Potassium Hydroxide V.S.,
 Cochineal or Iodeosin T.S., each, *a sufficient quantity*.

Place the Coca in an Erlenmeyer flask, add 50 Cc. of a mixture of chloroform 1 volume and ether 4 volumes, and insert the stopper securely. Allow the flask to stand ten minutes, then add 2 Cc. of ammonia water mixed with 3 Cc. of distilled water, and shake the flask well, at frequent intervals, during one hour. Then transfer as much as possible of the contents of the flask to a small percolator which has been provided with a pledget of cotton packed firmly in the neck, and inserted in a separator containing 6 Cc. of normal sulphuric acid V.S., diluted with 20 Cc. of distilled water. When the liquid has passed through the cotton, pack the Coca firmly in the percolator with the aid of a glass rod, and, having rinsed the flask with 10 Cc. of chloroform-ether mixture, transfer the remaining contents of the flask to the percolator by the aid of several small portions (5 Cc.) of a chloroform-ether mixture, using the same proportions as before, and continue the percolation with successive small portions of the same liquid (in all 50 Cc.). Next, shake the separator well for one minute, after securely inserting the stopper, and when the liquids have completely separated, draw off the acid liquid into another separator. Add to the chloroform-ether mixture 10 Cc. of a sulphuric acid mixture, using the same proportions as before, agitate well, and again draw off the acid liquid. Repeat this operation once more, drawing off the acid solution as before into the second separator, introduce a small piece of red litmus-paper, add ammonia water until the liquid is distinctly alkaline,

and shake out with 3 successive portions of ether (25, 20, and 15 Cc.). Collect the ether solutions in a beaker, place it on a water-bath filled with warm water, and allow the ether to evaporate entirely. Dissolve the residue in 3 Cc. of ether, and let this also evaporate completely. To the alkaloidal residue add 4 Cc. of tenth-normal sulphuric acid V.S. and 5 drops of cochineal or iodeosin T.S., then titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 4 (the 4 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.03, and this product by 10, to obtain the percentage of ether-soluble alkaloids contained in the Coca.

ASSAY OF FLUIDEXTRACT OF COCA

Recipe.—Fluidextract of Coca, *ten cubic centimeters*. 10 Cc.

Ammonia Water,
Ether,
Distilled Water,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S. or Iodeosin T.S., each, *a sufficient quantity*.

Transfer 10 Cc. of Fluidextract of Coca by means of a graduated pipette to a separator, add 25 Cc. of ether, and then 2 Cc. of ammonia water, shaking together for one minute. When the liquids have completely separated, draw off the lower aqueous layer into a second separator, and to this add 20 Cc. more of ether, and repeat the shaking for one minute. Draw off and reject the lower aqueous layer from the second separator, and add the ether layer to the first separator. To this separator now add 5 Cc. of normal sulphuric acid V.S. and 5 Cc. of distilled water, and shake it well for one or two minutes. After the liquids have separated, draw off the lower aqueous layer into the other separator, and repeat the extraction in the first separator with 9 Cc. of distilled water and 1 Cc. of normal sulphuric acid V.S., shaking the liquids for one minute, and separating as before. Add the aqueous solution to the other separator and reject the ether. Now add to the combined acid liquids 20 Cc. of ether, a small piece of red litmus-paper, and sufficient ammonia water to render the mixture distinctly alkaline, and shake the liquids for one or two minutes. Draw off the separated aqueous layer into the other separator and the ether layer into a beaker. Repeat the extraction of the aqueous layer in the other separator with two portions (15 Cc. each) of ether, and add the resulting ether solutions to the beaker. Now evaporate the ether from the beaker, and, when dry, add to it 5 Cc. of tenth-normal sulphuric acid V.S., and stir until the alkaloidal residue is dissolved. Then add 5 drops of cochineal T.S. or iodeosin T.S., and titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 5 (the 5 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.03, and this product by 10, to obtain the weight in grammes of ether-soluble alkaloids contained in *one hundred cubic centimeters* of the Fluidextract of Coca.

ASSAY OF HYOSCYAMUS

The method to be employed is identical with that given for Belladonna Leaves (p. 1028), with the exception that *twenty-five grammes* of Hyoscyamus in No. 60 powder are to be used, the quantity of chloroform-ether mixture which is added at first increased from 50 Cc. to 100 Cc., and the product at the end of the assay multiplied by 4 instead of 10.

ASSAY OF EXTRACT OF HYOSCYAMUS

The method to be employed is identical with that given on p. 1030, using *ten grammes* of Extract of Hyoscyamus instead of the quantity of Extract of Belladonna Leaves there directed, and multiplying the product by 10 instead of 20.

ASSAY OF FLUIDEXTRACT OF HYOSCYAMUS

The method to be employed is identical with that given on p. 1031, using *fifty cubic centimeters* of Fluidextract of Hyoscyamus instead of the quantity of Fluidextract of Belladonna Root there directed, and multiplying the product by 2 instead of 10.

ASSAY OF TINCTURE OF HYOSCYAMUS

Transfer 100 Cc. of Tincture of Hyoscyamus to an evaporating dish, and evaporate it on a water-bath until it measures about 10 Cc. Add, if necessary, sufficient alcohol to dissolve any separated substance, and then assay the resulting liquid by the method given under *Fluidextractum Belladonnae Radicis* (p. 1031), using the same details as there directed for 10 Cc. of Fluidextract of Belladonna Root, with the exception that the multiplication by 10 be omitted; the result will represent the weight in grammes of alkaloids contained in *one hundred cubic centimeters* of Tincture of Hyoscyamus.

ASSAY OF IPECAC

Recipe.—Ipecac, in No. 80 powder, fifteen grammes..... 15 Gm.

Ether,
Chloroform,
Ammonia Water,
Distilled Water,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S., a sufficient quantity.

Introduce the Ipecac into an Erlenmeyer flask of 250-Cc. capacity, add 115 Cc. of ether and 35 Cc. of chloroform, shake the flask during five minutes, and then add 3 Cc. of ammonia water and again shake the flask at intervals during half an hour. Now add 10 Cc. of distilled water, shake the liquid until the powder collects in masses, and pour off 100 Cc. of the clear ethereal solution into a measuring cylinder. Transfer the latter to a separator, add 10 Cc. of normal sulphuric acid V.S. and 10 Cc. of distilled water. Shake the separator moderately during two minutes, and when the liquids have separated, draw off the lower acid solution into a second separator. Repeat the shaking out of the ether solution with 3 Cc. of normal sulphuric acid V.S. and 5 Cc. of distilled water, drawing the acid solution into the second separator. Repeat the shaking out again, using 10 Cc. of distilled water, and add the aqueous solution to the second separator. Reject the ether in the first separator, introduce a small piece of red litmus-paper into the second separator, add enough ammonia water to render the liquid alkaline and 25 Cc. of ether, and then shake the separator vigorously during one minute; draw off the alkaline aqueous liquid into another separator and transfer the ether solution to a flask. Add 20 Cc. of ether to the alkaline liquid in the separator, shake it for one minute, and, having allowed the liquids to separate, draw off the alkaline liquid into the other separator, and transfer the ether solution to the flask. Again shake out the alkaline liquid with 10 Cc. of ether, and, when the fluids have separated, reject the alkaline liquid and add the ether solution to the liquid in the flask. Distil the ether from the flask with the aid of a water-bath, and dissolve the alkaloidal residue in 12 Cc. of tenth-normal sulphuric acid V.S., warming it gently on a water-bath if necessary. Then add five drops of cochineal T.S. and titrate with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 12 (the 12 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0238, and this product by 10, which will give the percentage of alkaloids in the Ipecac.

ASSAY OF FLUIDEXTRACT OF IPECAC

Recipe.—Fluidextract of Ipecac, ten cubic centimeters..... 10 Cc.

Ammonia Water,
Ether,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S., each, a sufficient quantity.

Transfer 10 Cc. of Fluidextract of Ipecac by means of a graduated pipette to a porcelain evaporating dish. Evaporate off the alcohol with the aid of a water-bath, and, when almost cool, add 5 Cc. of normal sulphuric acid V.S., and 20 Cc. distilled water, and stir the liquid at intervals for three minutes. Filter the liquid into a separator, rinse the dish, and wash the filter successively with 10 Cc. and 5 Cc. of

distilled water, and add these liquids to the separator. To the separator add 20 Cc. of ether and a small piece of red litmus-paper; render the liquid alkaline with ammonia water and shake the separator for one minute. Draw off the aqueous layer into a beaker and the ether layer into another beaker. Return the aqueous solution to the separator, add 10 Cc. more of ether, and shake the liquid, adding the ether solution to that already in the beaker, and returning the aqueous solution to the separator; repeat the extraction with 10 Cc. more of ether, and then add the ether layer to that already in the beaker. Allow the combined ether solutions to evaporate, either spontaneously or with the aid of a water-bath containing warm water, and then add 10 Cc. of tenth-normal sulphuric acid V.S. Stir the liquid carefully with a glass rod to facilitate the solution of the alkaloids, and when these have all dissolved, add 5 drops of cochineal T.S. From a graduated burette add sufficient fiftieth-normal potassium hydroxide V.S. until a pink color is produced. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 10 (the 10 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0238, and this product by 10, which will give the weight in grammes of alkaloids contained in each one hundred cubic centimeters of Fluidextract of Ipecac.

ASSAY OF NUX VOMICA

Recipe.—Nux Vomica, in No. 60 powder, twenty grammes.....20 Gm.

Ammonia Water,

Ether,

Chloroform,

Alcohol,

Normal Sulphuric Acid V.S.,

Fiftieth-normal Potassium Hydroxide V.S.,

Nitric Acid (sp. gr. 1.42),

Sodium Hydroxide Solution (1 in 10),

Tenth-normal Sulphuric Acid V.S.,

Sulphuric Acid Solution (3 per cent. H_2SO_4),

Iodeosin T.S. each, a sufficient quantity.

Introduce the Nux Vomica into a 250-Cc. Erlenmeyer flask and add to it 200 Cc. of a mixture of 137.5 Cc. of ether, 44 Cc. of chloroform, 13.5 Cc. of alcohol, and 5 Cc. of ammonia water; insert the stopper securely and macerate with frequent shaking during one hour and allow it to stand in a cool place for twelve hours. Decant into a measuring cylinder 100 Cc. of the liquid (representing 10 Gm. of Nux Vomica), and pour this into a separator, preferably of a globular shape. Rinse the cylinder with a little chloroform, add this to the separator, and then add 15 Cc. of normal sulphuric acid V.S.; shake the mixture moderately during one minute, being careful to avoid emulsification; when the liquids have separated completely, draw off the acid liquid into a beaker. Repeat the shaking out with successive portions of 5 and 3 Cc. of normal sulphuric acid V.S.; collect the acid solutions and pour them into a separator. If a drop of the last acid solution yields a precipitate with mercuric potassium iodide T.S., repeat the shaking out of the ether solution with 5 Cc. of normal sulphuric acid V.S. To the combined acid solutions in the separator, add a small piece of red litmus-paper, 25 Cc. of chloroform, and then sufficient ammonia water to render the liquid alkaline, and shake the separator thoroughly. When the liquids have separated, draw off the chloroform into a flask of 100-Cc. capacity, and repeat the shaking out of the alkaline liquid with two successive portions of 15 Cc. each of chloroform, adding the latter to that already in the flask. Evaporate the combined chloroformic solutions in the flask until the alkaloidal residue is dry, then dissolve it in 15 Cc. of sulphuric acid (3 per cent.), warming it on a water-bath. When the solution has cooled, add 3 Cc. of a cooled mixture of equal volumes of nitric acid (specific gravity 1.42) and distilled water, and after rotating the liquid a few times, set it aside for exactly ten minutes, shaking it gently three times during this interval. Transfer the resulting red liquid to a separator containing 25 Cc. of an aqueous solution of sodium hydroxide (1 in 10), and wash the flask three times with very small amounts of distilled water, and add the washings to the separator. If the liquid is not turbid, add 2 Cc. more of the solution of sodium hydroxide. Now add 20 Cc. of chloroform to the separator, and shake it well by a rotating motion for a few minutes, allow the liquids to separate, and draw off the chloroform through a small filter, wetted with chloroform, into a flask. Repeat this twice, using 10 Cc.

of chloroform each time, and draw off both portions into the flask, using the same filter. Finally, wash the filter and funnel with 5 Cc. of chloroform, and then evaporate all the chloroform by means of a water-bath very carefully, to avoid decrepitation. To the alkaloidal residue add 6 Cc. of tenth-normal sulphuric acid V.S., 5 drops of iodeosin T.S., about 80 Cc. of distilled water, and 20 Cc. of ether. When all the alkaloid is dissolved, titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. until the aqueous liquid just turns pink. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 6 (the 6 Cc. of tenth-normal sulphuric acid V.S. taken), multiply the remainder by 0.0332, and this product by 10, which will give the percentage of strychnine in the *Nux Vomica*.

ASSAY OF EXTRACT OF NUX VOMICA

Recipe.—Extract of *Nux Vomica*, two grammes. 2 Gm.

Distilled Water,
Ether,
Chloroform,
Ammonia Water,
Sulphuric Acid Solution (3 per cent. H_2SO_4),
Nitric Acid, (sp. gr. 1.42),
Sodium Hydroxide Solution (1 in 10),
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Iodeosin T.S., each, a sufficient quantity.

Introduce the Extract of *Nux Vomica* into a beaker, and dissolve it in 25 Cc. of a mixture of 16 Cc. of ether, 5 Cc. of chloroform, and 4 Cc. of ammonia water. When dissolved, transfer it to a separator, rinsing the beaker with a little chloroform, and adding the rinsings to the separator. Insert the stopper securely and shake the separator carefully for a few minutes. Draw off the aqueous layer into another separator, washing the ether solution and separator with a little water, and adding this to the second separator. Then shake out the aqueous liquid with two portions of 15 and 10 Cc., respectively, of chloroform, and add these to the first separator. If a few drops of the liquid left in the second separator still give a reaction with mercuric potassium iodide T.S. after acidulating, repeat the shaking out with 10 Cc. more of chloroform. Now shake out the chloroformic solution in the first separator with three portions of 15, 10, and 10 Cc. of sulphuric acid solution (3 per cent.), and collect the combined acid solutions in another separator. Introduce a small piece of red litmus-paper, add enough ammonia water to render the liquid alkaline, and extract the mixture with three portions, respectively, of 15, 10, and 10 Cc. of chloroform. Draw off the chloroformic solutions into a beaker and evaporate the chloroform with the aid of a water-bath. Dissolve the alkaloidal residue in the beaker in 15 Cc. of 3 per cent. sulphuric acid solution by the aid of a water-bath, and allow the liquid to cool. To this solution add 3 Cc. of a cooled mixture of equal volumes of nitric acid (specific gravity 1.42) and distilled water, and after rotating the liquid a few times, set it aside for exactly ten minutes, stirring it gently three times during this interval. Transfer the resulting red liquid to a separator containing 25 Cc. of an aqueous solution of sodium hydroxide (1 in 10), and wash the beaker three times with very small amounts of distilled water, and add the washings to the separator. If the liquid is not quite turbid, add 2 Cc. more of the solution of sodium hydroxide. Now add 20 Cc. of chloroform to the separator, and shake it well by a rotating motion for a few minutes, allow the liquids to separate, and draw off the chloroform through a small filter, wetted with chloroform, into a flask. Repeat this twice, using 10 Cc. of chloroform each time, and draw off both portions into the flask, using the same filter. Finally, wash the filter and funnel with 5 Cc. of chloroform, and then evaporate all the chloroform by means of a water-bath, very carefully, to avoid decrepitation. To the alkaloidal residue add 10 Cc. of tenth-normal sulphuric acid V.S., 5 drops of iodeosin T.S., about 90 Cc. of distilled water, and 20 Cc. of ether. When all the alkaloid is dissolved, titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. until the aqueous liquid just turns pink. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract this number from 10 (the 10 Cc. of tenth-normal sulphuric acid V.S. taken), multiply the remainder by 0.0332, and this product by 50, which will give the percentage of strychnine contained in the Extract of *Nux Vomica*.

ASSAY OF FLUIDEXTRACT OF NUX VOMICA

Recipe.—Fluidextract of Nux Vomica, *ten cubic centimeters*. . 10 Cc.

Ammonia Water,
Ether,
Chloroform,
Distilled Water,
Normal Sulphuric Acid V.S.,
Sulphuric Acid Solution (3 per cent. H_2SO_4),
Sodium Hydroxide Solution (1 in 10),
Nitric Acid (sp. gr. 1.42),
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Iodeosin T.S., each, *a sufficient quantity*.

Transfer 10 Cc. of Fluidextract of Nux Vomica by means of a graduated pipette to a porcelain dish, evaporate it to dryness with the aid of a water-bath, and dissolve the residue, while warm, in a mixture of 16 Cc. of ether, 5 Cc. of chloroform, and 4 Cc. of ammonia water, and transfer the solution to a separator, rinsing the dish with a little chloroform, which is to be added to the separator, and shake the separator carefully for a few minutes. When the fluids have separated, draw off the aqueous layer into another separator, wash the chloroform-ether liquid and separator with a little water, and add this to the second separator. Then shake the aqueous liquid with two successive portions of 15 and 10 Cc., respectively, of chloroform, and add these to the first separator. If a small portion of the liquid left in the second separator still shows, after acidifying, a reaction with mercuric potassium iodide T.S., repeat the shaking out with 10 Cc. more of chloroform. Now shake the combined liquids in the first separator with three successive portions, respectively, of 15, 10, and 10 Cc. of normal sulphuric acid V.S., and collect the combined acid solutions in another separator. To this acid solution add a small piece of red litmus-paper, and sufficient ammonia water to render it alkaline, then shake out successively with three portions, respectively, of 25, 10, and 10 Cc. of chloroform, and collect the chloroform solutions in a beaker. Evaporate the chloroform with the aid of a water-bath, dissolve the alkaloidal residue in 15 Cc. of 3 per cent. sulphuric acid solution, by the aid of a water-bath, and allow the liquid to cool. To this solution add 3 Cc. of a cooled mixture of equal volumes of nitric acid (specific gravity 1.42) and distilled water, and, after rotating the liquid a few times, set it aside for exactly ten minutes, stirring it gently three times during this interval. Transfer the resulting red liquid to a separator containing 25 Cc. of an aqueous solution of sodium hydroxide (1 in 10), and wash the beaker three times with very small amounts of distilled water, and add the washings to the separator. If the liquid is not quite turbid, add 2 Cc. more of the solution of sodium hydroxide. Now add 20 Cc. of chloroform to the separator, and shake it well by a rotating motion for a few minutes, allow the liquids to separate, and draw off the chloroform through a small filter, wetted with chloroform, into a flask. Repeat this twice, using 10 Cc. of chloroform each time, and draw off both portions into the flask, using the same filter. Finally, wash the filter and funnel with 5 Cc. of chloroform, and then evaporate all the chloroform by means of a water-bath, very carefully, to avoid decrepitation. To the alkaloidal residue add 10 Cc. of tenth-normal sulphuric acid V.S., 5 drops of iodeosin, T.S., about 80 Cc. of distilled water, and 20 Cc. of ether. When all the alkaloid is dissolved, titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. until the aqueous liquid just turns pink. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. taken by 5, subtract this number from 10 (the 10 Cc. of tenth-normal sulphuric acid V.S. taken), multiply the remainder by 0.0332, and this product by 10, which will give the weight in grammes of strychnine contained in *one hundred cubic centimeters* of the Fluidextract of Nux Vomica.

ASSAY OF TINCTURE OF NUX VOMICA

Transfer 100 Cc. of Tincture of Nux Vomica to a porcelain dish, evaporate it to dryness on a water-bath, and assay the resulting extract by the method given under *Extractum Nucis Vomicae* (p. 1035), using the same details as there directed for 2 Gm. of Extract of Nux Vomica, with the exception that the multiplication by 50 be omitted; the result will represent the weight in grammes of strychnine contained in *one hundred cubic centimeters* of Tincture of Nux Vomica.

ASSAY OF PHYSOSTIGMA

Recipe.—Physostigma, in No. 60 powder, twenty grammes. 20 Gm.

Ether,
Solution of Sodium Bicarbonate (1 in 20),
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Distilled Water,
Iodeosin T.S., each, a sufficient quantity.

Introduce the Physostigma into an Erlenmeyer flask of about 250-Cc. capacity, add 200 Cc. of ether, and shake the flask well during ten minutes. Then add 10 Cc. of an aqueous solution of sodium bicarbonate (1 in 20), and shake the mixture vigorously at intervals during four hours. Allow the powder to settle, and decant 100 Cc. of the ether solution (representing 10 Gm. of Physostigma) into a measuring cylinder; then transfer it to a separator, introduce a small piece of blue litmus-paper, and add sufficient normal sulphuric acid V.S. to render the liquid acid, and then 10 Cc. of distilled water. Shake the liquid well for several minutes, and draw off the aqueous layer into another separator. Repeat the extraction, using 2 Cc. of normal sulphuric acid V.S. and 8 Cc. of distilled water, add the acid aqueous layer to the second separator, and finally again shake out the ether solution, using 1 Cc. of normal sulphuric acid V.S. and 9 Cc. of distilled water, adding this also to the second separator. To the combined acid liquids in the second separator add 25 Cc. of ether, a small piece of red litmus-paper, and sufficient sodium bicarbonate solution (1 in 20) to render it alkaline. Shake the separator for one minute, allow the liquids to separate, and draw off the ether into a beaker. Repeat the shaking out process with 20 Cc. and again with 15 Cc. of ether added to the separator, shake each time for one minute, allow the liquids to separate, and draw off the ether into the beaker. Carefully evaporate the ether from the combined solutions by means of a water-bath, and when dry, dissolve the residue in 5 Cc. of tenth-normal sulphuric acid V.S. and 20 Cc. of ether, which must be strictly neutral, and transfer this solution to a bottle, rinsing with 80 Cc. of water; add 5 drops of iodeosin T.S., and titrate the excess acid with fiftieth-normal potassium hydroxide V.S., until, after shaking, the aqueous liquid just acquires a pink color. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 5 (the 5 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0273, and this product by 10; the result will be the percentage of alkaloids soluble in ether contained in the Physostigma. The figure 0.0273 represents the weight in grammes of alkaloids (mainly physostigmine) required to neutralize 1 Cc. of tenth-normal sulphuric acid V.S.

ASSAY OF EXTRACT OF PHYSOSTIGMA

Recipe.—Extract of Physostigma, one gramme. 1 Gm.

Alcohol,
Diluted Alcohol,
Ether,
Sodium Bicarbonate,
Normal Sulphuric Acid V.S.,
Distilled Water,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Iodeosin T.S., each, a sufficient quantity.

Transfer the Extract of Physostigma to a small porcelain dish, add 5 Cc. of diluted alcohol, and digest for five minutes in a water-bath below boiling temperature; then add about 5 Gm. of very clean, fine quartz sand, and evaporate to dryness on a water-bath, triturating thoroughly with a pestle to secure uniform admixture. When dry, carefully transfer the contents of the dish to an Erlenmeyer flask, add a little more sand to the dish, and by rubbing remove any adherent extract, which transfer to the flask, add 100 Cc. of ether, and shake the flask. Then add 10 Cc. of an aqueous solution of sodium bicarbonate (1 in 20) and shake the contents vigorously at intervals for one hour. Allow the mixture to stand, and, when settled, decant 50 Cc. of the ether solution into a separator, to which add a small piece of blue litmus-paper, sufficient normal sulphuric acid V.S. to render the liquid acid, and 10 Cc. of distilled water.

Shake the separator well for one minute, and draw off the aqueous layer into another separator. Repeat the shaking-out process, using 2 Cc. of normal sulphuric acid V.S. and 8 Cc. of distilled water, and add the acid aqueous layer to the second separator; again repeat the extraction, using 1 Cc. of normal sulphuric acid V.S. and 9 Cc. of distilled water, and add this to the second separator. To the combined acid liquids in the second separator add 25 Cc. of ether, a small piece of red litmus-paper, and sufficient sodium bicarbonate solution (1 in 20) to render it alkaline. Shake the separator for one minute, allow the liquids to separate, and draw off the ether into a beaker. Repeat the shaking-out process with 20 Cc. and again with 15 Cc. of ether added to the separator; shake each time for one minute, allow the liquids to separate, and draw off the ether into the beaker. Carefully evaporate the ether from the combined solutions by means of a water-bath, and, when dry, dissolve the residue in 2 Cc. of tenth-normal sulphuric acid V.S.; rinse the solution carefully into a 200-Cc. flask with distilled water, add enough distilled water to bring the volume to about 90 Cc., add 25 Cc. of ether, and having shaken the flask, add 5 drops of iodeosin T.S., then titrate the excess of acid with fiftieth-normal potassium hydroxide V.S., until, after shaking, the aqueous liquid just acquires a pink color. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 2 (the 2 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.0273, and this product by 200; the result will be the percentage of ether-soluble alkaloids contained in the Extract of Physostigma.

ASSAY OF TINCTURE OF PHYSOSTIGMA

Transfer 100 Cc. of Tincture of Physostigma to a porcelain dish, evaporate it to dryness on a water-bath, and assay the resulting extract by the method given under *Extractum Physostigmatis* (just above), using the same details as there directed for 1 Gm. of Extract of Physostigma, with the exception that the product must be multiplied by 2 instead of 200; the result will represent the weight in grammes of ether-soluble alkaloids from Physostigma contained in *one hundred cubic centimeters* of Tincture of Physostigma.

ASSAY OF PILOCARPUS

Recipe.—Pilocarpus, in No. 60 powder, ten grammes. 10 Gm.

Chloroform,
Ammonia Water,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S. or Iodeosin T.S., each, a sufficient quantity.

Moisten the Pilocarpus with 2 Cc. of ammonia water and 3 Cc. of chloroform, and at once pack it firmly in a small cylindrical percolator, which has been provided with a pledget of cotton packed firmly in the neck. Percolate the powder slowly with chloroform containing about 2 per cent. of ammonia water, until it is exhausted, about 100 Cc. of menstruum usually being sufficient. Pour into a separator the percolate, and shake it out with 15 Cc. of normal sulphuric acid V.S., transferring the acid aqueous layer to another separator, and repeating the shaking out of the chloroform solution with 2 Cc. of normal sulphuric acid V.S., mixed with 8 Cc. of distilled water. Add the acid layer to the second separator, and again repeat the shaking out with 10 Cc. of distilled water, and add the aqueous liquid to the second separator. Introduce into the second separator a small piece of red litmus-paper, add enough ammonia water to render the liquid alkaline, and shake out the liquid with 20 Cc. of chloroform, drawing off the chloroformic solution into a beaker. Repeat the shaking out with two portions of 15 and 10 Cc. each of chloroform, and add the chloroformic solutions to the beaker. Evaporate the chloroform by means of a water-bath, and dissolve the alkaloidal residue in 7 Cc. of tenth-normal sulphuric acid V.S. Add 5 drops of cochineal T.S. or iodeosin T.S., and titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 7 (the 7 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.02, and this product by 10; the result will be the percentage of alkaloids contained in the Pilocarpus. The figure 0.02 represents the weight in grammes of alkaloids (mainly pilocarpine) required to neutralize 1 Cc. of tenth-normal sulphuric acid V.S.

ASSAY OF FLUIDEXTRACT OF PILOCARPUS

Recipe.—Fluidextract of Pilocarpus, *ten cubic centimeters*.... 10 Cc.
Chloroform,
Ammonia Water,
Normal Sulphuric Acid V.S.,
Tenth-normal Sulphuric Acid V.S.,
Fiftieth-normal Potassium Hydroxide V.S.,
Cochineal T.S. or Iodeosin T.S., each, *a sufficient quantity*.

Transfer 10 Cc. of Fluidextract of Pilocarpus by means of a graduated pipette to a porcelain dish containing a little clean sand, and evaporate it to dryness with the aid of a water-bath. Mix the sand uniformly with the extract and transfer the mixture to an Erlenmeyer flask of about 100-Cc. capacity, rinsing the dish with a mixture of 25 Cc. of chloroform and 2.5 Cc. of ammonia water. Transfer the rinsings to the flask, cork it securely, and shake it well at intervals during one hour. Decant the liquid, transfer to a separator, wash the sand with several portions of chloroform, draw off and filter the chloroformic liquid into another separator. Then shake out the chloroform solution with 15 Cc. of normal sulphuric acid V.S., transferring the acid aqueous solution to another separator. Repeat the shaking out with a mixture of 5 Cc. of normal sulphuric acid V.S. and 5 Cc. of distilled water, collecting the acid solutions in the second separator. Again repeat the shaking out with 10 Cc. of distilled water, and add the aqueous liquid to the second separator. Introduce into the second separator a small piece of red litmus-paper, add enough ammonia water to render the liquid alkaline, and shake out the liquid with 20 Cc. of chloroform, drawing off the chloroformic solution into a beaker. Repeat the shaking out with two portions of 15 and 10 Cc. each of chloroform, and add the chloroformic solutions to the beaker. Evaporate the chloroform by means of a water-bath, and dissolve the alkaloidal residue in 8 Cc. of tenth-normal sulphuric acid V.S. Add 5 drops of cochineal T.S. or iodeosin T.S., and titrate the excess of acid with fiftieth-normal potassium hydroxide V.S. Divide the number of cubic centimeters of fiftieth-normal potassium hydroxide V.S. used by 5, subtract the quotient from 8 (the 8 Cc. of tenth-normal sulphuric acid V.S. taken), and multiply the remainder by 0.02, and this product by 10, to obtain the weight in grammes of alkaloids contained in *one hundred cubic centimeters* of the Fluidextract of Pilocarpus.

ASSAY OF SCOPOLA

The method to be employed is identical with that given for Belladonna Leaves, on p. 1028, using *ten grammes* of Scopola, in No. 60 powder.

ASSAY OF EXTRACT OF SCOPOLA

The method to be employed is identical with that given on p. 1030, using *two grammes* of Extract of Scopola instead of the quantity of Extract of Belladonna Leaves there directed. The product must be multiplied by 50 instead of 20.

ASSAY OF FLUIDEXTRACT OF SCOPOLA

The method to be employed is identical with that given for Fluidextract of Belladonna Root on p. 1031, using *ten cubic centimeters* of Fluidextract of Scopola.

ASSAY OF STRAMONIUM

The method to be employed is identical with that given on p. 1028 for Belladonna Leaves, using *ten grammes* of Stramonium, in No. 60 powder.

ASSAY OF EXTRACT OF STRAMONIUM

The method to be employed is identical with that given for Extract of Belladonna Leaves on p. 1030, using *five grammes* of Extract of Stramonium.

ASSAY OF FLUIDEXTRACT OF STRAMONIUM

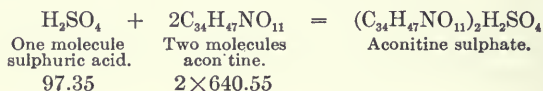
The method to be employed is identical with that given for Fluidextract of Belladonna Root on p. 1031, using *ten cubic centimeters* of Fluidextract of Stramonium.

ASSAY OF TINCTURE OF STRAMONIUM

Transfer 100 Cc. of Tincture of Stramonium to an evaporating dish, and evaporate it on a water-bath until it measures about 10 Cc. Add, if necessary, sufficient alcohol to dissolve any separated substance, and then assay the resulting liquid by the method given under *Fluidextractum Belladonnæ Radicis* (p. 1031), using the same details as there directed for 10 Cc. of Fluidextract of Belladonna Root, with the exception that the multiplication by 10, as there directed, be omitted; the result will represent the weight in grammes of alkaloids contained in one hundred cubic centimeters of Tincture of Stramonium.

Like the assays by weight, these assays by titration defy both condensation and tabulation. It may be well, however, to consider for a few moments the arithmetic of the titration.

Taking the assay of aconite as a type, it will be well to notice that the equation used in calculation of the amount of aconitine in the drug, the fluidextract and the tincture contains as constant factor 0.064. This, it will be noted, is the molecular weight of aconitine (p. 915) divided by 10,000. It will also be noted that this factor, 0.064, is the amount of aconitine represented by 1 Cc. tenth-normal sulphuric acid V.S. (p. 991), this simply following the regular process of finding the equivalent value for that amount of volumetric solution. Repeating the process in this special case:



Remembering (p. 991) that 1 Cc. tenth-normal $\text{H}_2\text{SO}_4 = 0.0048675$ Gm. absolute H_2SO_4 , we see—

$$\begin{aligned} \text{That } 97.35 \text{ Gm. absolute } \text{H}_2\text{SO}_4 &= 2 \times 640.55 \text{ Gm. aconitine,} \\ \text{that } 48.675 \text{ Gm. absolute } \text{H}_2\text{SO}_4 &= 640.55 \text{ Gm. aconitine.} \\ \text{And that } 0.0048675 \text{ Gm. absolute } \text{H}_2\text{SO}_4 &= \frac{1}{10,000} \times 640.55 \text{ Gm. aconitine.} \\ &\text{or } 0.064 \text{ Gm. aconitine.} \end{aligned}$$

Taking up the difference in the equation of calculation of the three forms of aconite (see pp. 1027 and 1028):

$$\left. \begin{array}{l} \text{Aconite (drug)} \\ \text{Fluidextract aconite} \end{array} \right\} \frac{(3 - \text{Cc. } \frac{1}{50}n \text{ KOH})}{5} \times 0.064 \times 10 = \text{per cent. aconitine.}$$

$$\text{Tincture aconite } \frac{(3 - \text{Cc. } \frac{1}{50}n \text{ KOH})}{5} \times 0.064 = \text{per cent. aconitine.}$$

We find that the figure 3 is the amount of tenth-normal sulphuric acid V.S. (3 Cc.) added to the freed alkaloids; that $\frac{\text{"Cc. } \frac{1}{50}n \text{ KOH"} }{5}$ means the number of cubic centimeters fiftieth-normal alkali divided by 5, thus expressing the alkali used in terms of tenth-normal solution; that 0.064 is the aconitine value of 1 Cc. of tenth-normal acid; and that multiplying by 10 is to find the amount of aconitine in 100 Gm. of the drug or 100 Cc. of the fluidextract. In calculating the strength of the tincture the 10 is omitted, since we start out with 100 Cc. of the tincture. Giving a concrete example, suppose 10 Gm. aconite is

VOLUMETRIC ASSAYS OF ORGANIC SUBSTANCES

extracted by official process and, mixed with 3 Cc. $\frac{1}{10}$ nH₂SO₄, 11 Cc. $\frac{1}{50}$ nKOH to neutralize excess of acid.

11 Cc. $\frac{1}{50}$ n KOH = $\frac{11}{5} = 2.2$ Cc. $\frac{1}{10}$ n KOH.
 2.2 Cc. $\frac{1}{10}$ n KOH = 2.2 Cc. $\frac{1}{10}$ n H₂SO₄.
 Then 2.2 Cc. $\frac{1}{10}$ n H₂SO₄ is left out of the 3 Cc. originally added.
 3 — 2.2 = 0.8 Cc. $\frac{1}{10}$ n H₂SO₄ combined with aconitine.
 1 Cc. $\frac{1}{10}$ n H₂SO₄ = 0.064 Gm. aconitine.
 So 0.8 Cc. $\frac{1}{10}$ n H₂SO₄ = 0.8 × 0.064 = 0.0512 Gm. aconitine.
 We see 10 Gm. aconite contains 0.0512 Gm. aconitine.
 Hence 100 Gm. aconite contains 10 × 0.0512 or 0.512 Gm. aconitine.

This method of reasoning applies to all of this class of assays; the definite numeric factor being the molecular weight of the appropriate alkaloid divided by 10,000; the amount of alkaloid (expressed in grammes) neutralized by one cubic centimeter of tenth-normal sulphuric acid V.S.

SPECIAL ACIDIMETRIC ASSAYS OF VOLATILE OILS

Two natural and two synthetic volatile oils, owing their activity to aldehydes, are assayed by the acidimetric process.

ASSAY OF BENZALDEHYDE

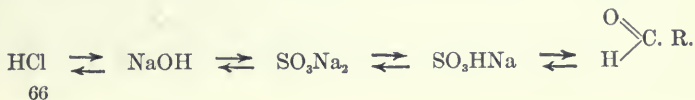
Introduce into a tared 150-Cc. flask 10 Cc. of purified kerosene, note the exact weight, add 12 drops of Benzaldehyde, and again note the weight; add 20 Cc. of distilled water with 6 drops of rosolic acid T.S., and then neutralize the solution exactly by the addition of tenth-normal sodium hydroxide V.S., agitating the flask thoroughly. Add from a burette, gradually, a solution of sodium sulphite (1 in 5), alternating with half-normal hydrochloric acid V.S. from a second burette, until 10 Cc. of the sodium sulphite solution have been added, and enough half-normal hydrochloric acid V.S. to maintain the neutrality of the mixture; after adding a few drops of rosolic acid T.S., and agitating the flask frequently, allow it to stand two hours to insure a permanent condition of neutrality, and then note the number of cubic centimeters of the half-normal hydrochloric acid V.S. used. Carry out a blank test, identical with the foregoing, except that the Benzaldehyde is omitted, and note the amount of half-normal hydrochloric acid V.S. consumed. Subtract the number of cubic centimeters required in the blank test from the number required in the original test; each Cc. of this difference corresponds to 0.0526 Gm. of Benzaldehyde. To find the percentage, multiply the above difference by 0.0526, and this product by 100, and divide the weight of the Benzaldehyde taken.

This assay is based on the fact that normal sodium sulphite in water may be regarded to change to a *very slight extent* in aqueous solutions as follows,



until an equilibrium is reached. If this equilibrium be disturbed by diluted HCl which combines with the NaOH, the change from normal to acid sulphite will continue.

This change is induced by aldehydes, which add on the elements of acid sulphite, forming hydroxysulphonates, etc. In this way we, therefore, titrate the NaOH formed by the interaction between aldehyde, normal sulphite, and water, computing the aldehyde directly:



Hence one molecule hydrochloric acid indirectly represents one molecule benzaldehyde.

Hence 36.18 Gm. absolute HCl = 105.25 Gm. absolute benzaldehyde,
 0.01809 Gm. absolute HCl $\left\{ = \frac{1809}{3618000} \times 105.25 \text{ or } 0.0526 \right.$
 (amount in 1 Cc. $\frac{1}{2}$ nHCl)
 Gm. absolute benzaldehyde.

Such is the way the factor 0.0526 is obtained, the rest of the calculation being based on the different amount of half-normal hydrochloric acid used in the benzaldehyde assay and in the blank test, and on the exact weight of the benzaldehyde employed.

Like benzaldehyde assay are the other three, viz.:

Oil of bitter almond, exactly the same, save 12 drops of the oil are used.

	AMOUNT SUB- STANCE.	AMOUNT WATER.	AMOUNT PHENOL- PHTHALEIN T.S.	AMOUNT Na ₂ SO ₃ SOLUTION.	LET STAND.	FACTOR.
<i>Cinnamic aldehyde.</i>	Twelve drops care- fully weighed di- rect (no kero- sene).	5 Cc.	Few drops.	50 Cc. ad- ded all at once.	In boiling water, adding half-nor- mal acid until neutral.	0.033
<i>Oil of Lemon (citral assay).</i>	15 Cc. carefully weighed direct (no kerosene).	5 Cc.	Few drops.	25 Cc. ad- ded all at once.		0.03802

SAPONIFICATION NUMBER OF OILS AND FATS

As already explained on p. 737, fixed oils and fats are esters of glycerin with fatty acids, and the process of soap-making is the cooking of such oils with soda or potassa, whereby the sodium or potassium salts of the fatty acids are obtained. This fact is utilized in the assay of oils and fats, a given weight of the fatty substance being boiled with a known excess of alcoholic potassium hydroxide T.S. (p. 994), and titrated with half-normal hydrochloric acid. In this way we can find out the exact amount of alkali taken up by the oil, and thus the "saponification number" can be deduced.

The pharmacopœial process is as follows:

Saponification Value of Fats and Oils.—The determination of the saponification value is conducted as follows: Weigh out accurately, in a flask holding 150 to 200 Cc., 1.5 to 2 Gm. of the purified and filtered fat. Next run into the flask, with a burette, 25 Cc. of alcoholic potassium hydroxide T.S. While exactly 25 Cc. is not indispensable, in comparative tests precisely the same amount must be used, allowing the burette to drain in exactly the same way in each test. Then place a small funnel in the flask and heat it on a water-bath containing boiling water, for half an hour, so that the alcohol is simmering, frequently imparting a rotatory motion to the contents of the flask. Then add 1 Cc. of phenolphthalein T.S., and titrate back the excess of potassium hydroxide with half-normal hydrochloric acid V.S. A blank test is made at the same time, using the alcoholic potassium hydroxide T.S. alone; the difference in the number of cubic centimeters of half-normal hydrochloric acid V.S. consumed by the blank test and the real test, multiplied by 27.87, and divided by the weight in grammes of the fat or oil, will give the saponification equivalent of the sample tested.

The saponification number means the number of milligrammes absolute potassium hydroxide which combines with 1 Gm. of oil. Thus, taking an example of calculating by process given above, suppose 2 Gm. of olive oil combines with 13.9 Cc. half-normal potas-

sium hydroxide V.S. That is, suppose it takes 25 Cc. half-normal hydrochloric acid V.S. required to neutralize 25 Cc. alcoholic potassa before saponification, and 11.1 Cc. half-normal hydrochloric acid V.S. to neutralize the 25 Cc. alcoholic potassa after saponification. The equations in this case would be $25 - 11.1 = 13.9$,

$$\frac{13.9 \times 27.87}{2} = \frac{390.104}{2} = 195.$$

and the saponification number is 195.

Now let us see the amount of potassa combining with the oil:

1 Cc. $\frac{1}{2}$ n KOH = 0.02787 Gm. absolute KOH (p. 995).
 13.9 Cc. $\frac{1}{2}$ n KOH = $13.9 \times 0.02787 = 0.390192$ Gm. absolute KOH.

Hence we see that 2 Gm. oil combines with 390 milligrammes KOH
 and 1 Gm. oil combines with 195 milligrammes KOH.

SAPONIFICATION NUMBER OF OFFICIAL OILS AND FATS

The following oils and fats are thus assayed:

	SAP. NO.		SAP. NO.
<i>Olive oil</i>	191-195	<i>Croton oil</i>	203-215
<i>Expressed oil of almond</i>	191-200	<i>Oil of theobroma</i>	188-195
<i>Cotton seed oil</i>	191-196	<i>Lard oil</i>	195-197
<i>Linseed oil</i>	187-195	<i>Cod liver oil</i>	175-185
<i>Castor oil</i>	179-183	<i>Yellow wax</i>	90- 96

The following volatile oils are assayed by saponification:

Oil of Rose.—Place in a weighing bottle about 2 Cc. of the Oil of Rose, and weigh it accurately. Transfer it, with the aid of a little alcohol, to a 100-Cc. flask, and add 20 Cc. of half-normal alcoholic potassium hydroxide V.S. Connect the flask with a reflux condenser, and boil the mixture during thirty minutes on a water-bath. When cool, add 50 Cc. of distilled water and a few drops of phenolphthalein T.S., and titrate with half-normal sulphuric acid V.S. Subtract the number of Cc. of half-normal sulphuric acid V.S. required from 20 (the 20 Cc. of half-normal alcoholic potassium hydroxide V.S. taken), multiply the difference by 27.87, and divide by the weight of the Oil to obtain the saponification value.

By this assay oil of rose should show a saponification value of not less than 10 nor more than 17; that is, each gramme of the oil should combine with 10 to 17 milligrammes absolute potassa. This saponification value is much smaller than that of the fixed oils, for while these are composed chiefly of ester, oil of rose contains but a small percentage of its peculiar ester, methyl ester of methyl anthranilic acid.

Oil of Peppermint.—Introduce into a tared flask 10 Cc. of Oil of Peppermint, and note the exact weight; add 25 Cc. of half-normal alcoholic potassium hydroxide V.S., connect with a reflux condenser, and boil the mixture during one hour. After cooling titrate the residual alkali with half-normal sulphuric acid V.S., using phenolphthalein T.S. as indicator. Subtract the number of cubic centimeters of half-normal sulphuric acid V.S. required from the 25 Cc. of half-normal alcoholic potassium hydroxide V.S. taken, multiply the difference by 9.834, and divide the product by the weight of the Oil of Peppermint taken to find the percentage of menthyl acetate. Wash the residual oil repeatedly with water, transfer it to a flask provided with

a ground-glass tube-condenser (acetylation flask), add 10 Cc. of acetic acid anhydride and about 1 Gm. of anhydrous sodium acetate, and boil gently during one hour. Allow it to cool, wash the acetylated oil with distilled water, and afterward with sodium hydroxide T.S., until the mixture is slightly alkaline to phenolphthalein T.S., and then dry it with the aid of fused calcium chloride, and filter.

Transfer to a tared 100-Cc. flask 5 Cc. of the dry acetylated oil, note the exact weight, add 50 Cc. of half-normal alcoholic potassium hydroxide V.S., connect with a reflux condenser, and boil the mixture during one hour. After cooling, titrate the residual alkali with half-normal sulphuric acid V.S., using phenolphthalein T.S. as indicator. Subtract the number of cubic centimeters of half-normal sulphuric acid V.S. required from 50 Cc. of half-normal alcoholic potassium hydroxide V.S. taken, multiply the difference by 7.749, and divide the product by the weight of the dry acetylated oil taken, less the above difference multiplied by 0.021; the quotient will represent the percentage of menthol in the Oil of Peppermint.

NOTE.—The difference referred to above represents the number of cubic centimeters of half-normal alcoholic potassium hydroxide V.S. consumed by the acetylated oil.

Oil of Rosemary.—Assayed like oil of peppermint, except the factor for the ester (bornyl acetate) is 9.734, and that for the alcohol (borneol) is 7.649. It will be noticed that both of these assays include two operations: the saponification of the characteristic ester by a process similar to that used in the assay of oil of rose, and then the assay of the characteristic alcohol by "acetylation."

As to the saponification tests, it will be seen that the factor (9.834 for the menthyl acetate of oil of peppermint; 9.734 for the bornyl acetate of oil of rosemary) represents the molecular weight of the characteristic ester divided by 200, or 100 times the amount of ester that combines with 1 Cc. half-normal potassium hydroxide V.S. (p. 995).

This can best be explained by a hypothetical assay.

Suppose we find 9 Gm. of oil of peppermint combines with 7.5 Cc. half-normal alcoholic KOH; that is, while the 25 Cc. half-normal alcoholic KOH took 25 Cc. half-normal H_2SO_4 before saponification, after saponification the alkali left took only 17.5 Cc. half-normal H_2SO_4 . In such cases we find—

that (a) 9 Gm. oil	= 7.5 Cc. $\frac{1}{10}$ n alcoholic KOH.
that (b) 1 Cc. $\frac{1}{10}$ n alcoholic KOH	= 0.09834 Gm. menthyl acetate.
that (c) 7.5 Cc. $\frac{1}{10}$ n alcoholic KOH	= 7.5×0.09834 .
	= 0.73755 Gm. menthyl acetate.
Therefore that (d) 9 Gm. oil	= 0.73755 Gm. menthyl acetate.
and that (e) 9 Gm. oil	= $\frac{9}{10} \times 0.73755 = 8.2$ per cent. menthyl acetate.

Careful examination of the above figures shows that the real calculation "c" and "e" means the multiplication of the number of cubic centimeters of alcoholic potassa used by 0.09834 and by 100 (or by 9.834), and finally dividing by weight of oil. In passing, it may be stated that the use of the acetylation factors (7.749 for the menthol in oil of peppermint and 7.649 for the borneol in oil of rosemary) can be explained in the same way.

The *acetylation tests* given above for oil of peppermint and for oil of rosemary, as well as that for the santalol in oil of santal, given below, are based on the fact that the characteristic alcohols of these three oils form well-defined acetyl compounds when boiled with acetic acid anhydride and sodium acetate, in the appropriate "acetylation

flask" (Fig. 231); that this oil, separated from the aqueous liquid, washed with water, and dried, can be saponified like any other ester, and from the amount of alcoholic potassa required for saponification the amount of alcohol can be estimated.

The oils thus assayed are:

Oil of Peppermint.—See p. 1043.

Oil of Rosemary.—See p. 1044.

Oil of Santal.—Like assay for menthol in oil of peppermint, except that 2 Gm. anhydrous sodium acetate are used; that the mixture is boiled in acetylation flask one and one-half hours; that 3 Cc. of the acetylated oil is taken for saponification; and that the factor is 11.026.

THE ACID NUMBER OF RESINS

As mentioned on p. 852, true resins contain resin acids. These resin acids, like other acids, combine with alkali in definite proportions. Hence their amount can be estimated by titration with volumetric alkali solution. The pharmacopœial directions for acid number estimations are as follows:

Resins, etc., Acid Number for.—Dissolve 1 Gm. of the resinous substance in alcohol, add a few drops of phenolphthalein T.S., and titrate with normal potassium hydroxide V.S.; the amount of potassium hydroxide consumed (expressed in milligrammes) is termed the Acid Number. The reaction is often more distinct if an excess of normal potassium hydroxide V.S. be used and the solution titrated back with normal acid V.S.

The official resins so tested are:

Guaiac (acid number 70 to 80); *mastic* (acid number not less than 65); *rosin* (acid number not less than 150).

IODINE ABSORPTION NUMBER.

As already explained on p. 628, fixed oils, like all other unsaturated bodies, when treated with halogens, notably iodine, loosen whatever double bonds they possess, attaching an atom of the halogen to each bond thus freed; hence each double bond in such oil takes up two atoms of iodine. As the percentage of unsaturated ester differs in each fixed oil, the different fixed oils absorb different amounts of iodine; hence the iodine absorption number is a valuable aid to identification and differentiation of oils. The pharmacopœial explanation of this test is as follows:

Iodine Absorption Value of Fats and Oils.—The iodine value or number of a fat or an oil is a figure which indicates the percentage of iodine absorbed under certain conditions. It is determined as follows: To a solution of 0.3 Gm.¹ of the fat or oil in 10 Cc. of chloroform contained in a glass-stoppered bottle of 250-Cc. capacity, add 25 Cc. of a mixture of equal volumes of alcoholic iodine T.S. and alcoholic mercuric chloride T.S., both of which have been measured from a burette. After having been securely stoppered, the bottle is set aside in a cool place, protected from the light, for a period of four² hours. After this time, the mixture must still

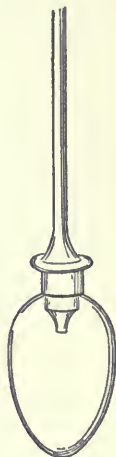


Fig. 231.—Acetylation flask.

¹ 0.15 to 0.2 Gm. for linseed oil and 0.8 Gm. for oil of theobroma and similar fats.

² Sixteen hours are required for accuracy in the case of linseed oil.

possess a brown color; if it does not, a further measured portion of the mixture of the two reagents should be added, and the mixture be again set aside. Finally, 20 Cc. of potassium iodide T.S. are added, followed by 50 Cc. of water, and tenth-normal sodium thiosulphate V.S. is then added in small successive portions, shaking thoroughly after each addition until the color of the mixture is discharged. The number of Cc. of the sodium thiosulphate V.S. consumed is noted. At the same time that this test is carried out, a blank experiment is made in which exactly the same quantities of chloroform, iodine T.S., and mercuric chloride T.S. are mixed, and after standing for four or more hours, the free iodine is estimated by titration with tenth-normal sodium thiosulphate V.S. as directed above. The number of Cc. of the thiosulphate V.S. consumed is noted, and from this is deducted the number of Cc. of the thiosulphate V.S. which was consumed in the test; the difference multiplied by 12.59, and this product divided by 3,³ gives the iodine value of the fat or oil.

Iodine Test Solution, Alcoholic.—Dissolve 25 Gm. of iodine [*Iodum*, U. S. P.] in 500 Cc. of alcohol. This solution is employed in the determination of the iodine absorption value of fats and oils.

Mercuric Chloride Test Solution, Alcoholic.—Dissolve 30 Gm. of mercuric chloride, HgCl_2 [*Hydrargyri Chloridum Corrosivum*, U. S. P.], in 500 Cc. of alcohol. This solution is employed in the determination of the iodine absorption value of fats and oils.

Explaining the arithmetic of this process, let us suppose that 0.3 Gm. of a certain fat plus 25 Cc. alcoholic iodine T.S. take 60 Cc. tenth-normal thiosulphate V.S. to decolorize, while in the blank experiment 25 Cc. alcoholic iodine T.S. takes 90 Cc. tenth-normal thiosulphate V.S. for decolorization. This would mean that 0.3 Gm. fat absorbed 90—60, or 30 Cc. tenth-normal thiosulphate V.S.

1 Cc. $\frac{1}{10}$ n thiosulphate V.S. = 0.01259 Gm. iodine (p. 1002).

30 Cc. $\frac{1}{10}$ n thiosulphate V.S. = 30×0.01259 Gm. iodine.
= 0.3777 Gm. iodine.

Hence 0.3 Gm. fat absorbed 0.3777 Gm. iodine.

Hence 100 Gm. fat would absorb $\frac{100.0}{0.3} \times 0.3777$ Gm. iodine
or 125.9 Gm. iodine.

The factor as given by the pharmacopœia is: "The number of Cc. thiosulphate consumed in the blank (90 Cc. in our case) is deducted from the number of centimeters thiosulphate consumed in the test (60 Cc. in our case), and the difference (30 in our case), multiplied by 12.59 and divided by 3, gives the iodine value."

$$\frac{30 \times 12.59}{3} = 125.9,$$

which shows that the so-called "iodine number" means *the number of grammes of iodine absorbed by 100 grammes of fat*.

The following official oils are tested exactly as in the process given above:

	IODINE VALUE.		IODINE VALUE.
<i>Olive oil</i>	80-88	<i>Croton oil</i>	103-109
<i>Expressed oil of almond</i>	95-100	<i>Lard oil</i>	56-74
<i>Cotton seed oil</i>	102-108	<i>Cod liver oil</i>	140-150

The following official oils deviate slightly from the general directions given above, the difference being noted in each case:

³ When the quantity of the fat or oil used is not 0.3 Gm., then the product is not divided by 3, but the figure corresponding to the quantity taken; thus, for linseed oil, 0.15 Gm. would be divided by 1.5.

<i>Linseed oil</i>	0.15 Gm. used; maceration is sixteen hours; iodine value at least 170.
<i>Castor oil</i>	0.3 Gm. used; maceration is eight hours; iodine value 84 to 89.
<i>Oil of theobroma</i>	0.8 Gm. used; maceration is four hours; iodine value 33 to 38.

SPECIAL ORGANIC VOLUMETRIC ASSAYS

I. PHENOL ASSAY WITH KOPPESCHAAR'S SOLUTION

As the theory of this assay is given on p. 760, it is merely necessary to insert pharmacopœial directions and to calculate results:

TENTH-NORMAL BROMINE VOLUMETRIC SOLUTION

[KOPPESCHAAR'S SOLUTION]

Br=79.36.

7.936 Gm. in 1000 Cc.

Dissolve 3.2 Gm. of potassium bromate and 50 Gm. of potassium bromide in sufficient water to measure, at or near 25° C. (77° F.), 900 Cc. Transfer 20 Cc. of this solution, by means of a pipette, into a bottle having a capacity of about 250 Cc. and provided with a glass stopper; add 75 Cc. of water and 5 Cc. of pure hydrochloric acid and immediately insert the stopper. Shake the bottle a few times, then remove the stopper just sufficiently to quickly introduce 5 Cc. of potassium iodide T.S., taking care that no bromine vapors escape, and immediately stopper the bottle. Agitate the bottle thoroughly, remove the stopper and rinse it and the neck of the bottle with a little water so that the washings flow into the bottle, and then add from a burette tenth-normal sodium thiosulphate V.S. until the brown iodine tint is just discharged. Note the number of Cc. of the sodium thiosulphate V.S. thus consumed, and then dilute the bromine solution so that equal volumes of it and of tenth-normal sodium thiosulphate V.S. will exactly correspond to each other under the conditions mentioned above.

EXAMPLE.—Assuming that the 20 Cc. of the bromine solution have required 25.2 Cc. of the sodium thiosulphate V.S. to completely discharge the iodine tint, then each 20 Cc. of the bromine solution must be diluted to 25.2 Cc. Thus, if 850 Cc. of the solution remain, it must be diluted with water to measure 1071 Cc.

After the solution is thus diluted, a new trial should be made in the manner above described, in which 25 Cc. of the tenth-normal sodium thiosulphate V.S. should just discharge the tint of the iodine liberated by the bromine set free from 25 Cc. of the standard bromine solution.

Keep the solution in dark amber-colored, glass-stoppered bottles.

One Cubic Centimeter of Tenth-Normal Bromine V.S. is the equivalent of:

	Gramme.
Bromine, Br.....	0.007936
Phenol, C ₆ H ₅ OH.....	0.001556

The only official chemical tested with this solution is—

ASSAY OF PHENOL

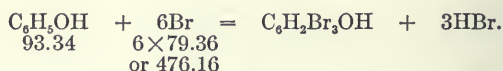
Dissolve 1.556 Gm. of the Phenol to be valued in a sufficient quantity of water to make 1000 Cc. Transfer 25 Cc. of this solution (containing 0.0389 Gm. of Phenol) to a glass-stoppered bottle having a capacity of about 200 Cc., add 30 Cc. of tenth-normal bromine V.S., then 5 Cc. of hydrochloric acid, and immediately insert the stopper. Shake the bottle repeatedly during half an hour, then remove the stopper just sufficiently to introduce quickly 5 Cc. of an aqueous solution of potassium iodide (1 in 5), being careful that no bromine vapor escapes, and immediately stopper the bottle. Shake the latter thoroughly, remove the stopper and rinse it and the neck of the bottle with a little water, so that the washings may flow into the bottle, and then add 1 Cc. of chloroform and shake well. Add, from a burette, tenth-normal sodium thiosulphate V.S., until the iodine tint is exactly discharged, and does not

reappear after thorough agitation. Note the number of Cc. of tenth-normal sodium thiosulphate V.S. consumed (which should not exceed 6 Cc.). The percentage of absolute Phenol is found by subtracting the number of Cc. of tenth-normal sodium thiosulphate V.S. used from 30 (the number of Cc. of bromine V.S. originally added), and multiplying the remainder by 4.

It will be seen that a certain amount of bromine is absorbed by the phenol; that the excess liberates iodine from the added potassium iodide, and that the iodine thus freed is titrated with tenth-normal thiosulphate solution.

We have already learned (p. 1000) that 1 Cc. tenth-normal thiosulphate V.S. equals 1 Cc. tenth-normal iodine V.S., and as the bromine liberates iodine atom for atom, 1 Cc. tenth-normal thiosulphate solution must equal 1 Cc. tenth-normal bromine solution.

We find above that 1 Cc. $\frac{1}{10}$ n bromine V.S. = 0.001556 Gm. phenol. This is because the reaction between bromine and phenol runs as follows:



We therefore see—

$$\begin{array}{lcl} 476.16 \text{ Gm. Br} & = & 93.34 \text{ Gm. C}_6\text{H}_5\text{OH.} \\ \text{Hence } 79.36 \text{ Gm. Br} = \frac{93.34}{6} & = & 15.56 \text{ Gm. C}_6\text{H}_5\text{OH,} \\ \text{and } 7.936 \text{ Gm. Br} & \} & \\ \text{or } 1000 \text{ Cc. } \frac{n}{10} \text{ Br V.S. } & \} & = 1.556 \text{ Gm. C}_6\text{H}_5\text{OH.} \\ \text{Hence } 1 \text{ Cc. } \frac{n}{10} \text{ Br V.S.} & = & 0.001556 \text{ Gm. CHOH.} \end{array}$$

In the pharmacopœial assay of phenol we take a solution equivalent to 0.0389 Gm. phenol, mix it with 30 Cc. tenth-normal bromine V.S., then with acid and potassium iodide, and finally titrate freed iodine with tenth-normal thio V.S. Suppose it takes 6 Cc. thiosulphate solution to decolorize, that would mean that there had been in the solution before adding the potassium iodide 6 Cc. tenth-normal bromine V.S., and that the phenol had combined with 30—6 to 24 Cc. tenth-normal bromine V.S.

$$\begin{array}{l} \text{Since } 1 \text{ Cc. } \frac{1}{10}\text{n Br V.S.} = 0.001556 \text{ Gm. phenol.} \\ 24 \text{ Cc. } \frac{1}{10}\text{n Br V.S.} = 24 \times 0.001556 \text{ Gm. phenol,} \\ \text{or } 0.037344 \text{ Gm. phenol.} \end{array}$$

Hence 0.0389 Gm. of the sample of phenol contained 0.037344 Gm. absolute phenol, or $\frac{0.037344}{0.038900}$ of 100 per cent. or 96 per cent. absolute phenol.

II. SUGAR ASSAY WITH FEHLING'S SOLUTION

This assay has been discussed on p. 695, and as neither of the official sugars is directed to be assayed, we can leave the subject after presenting pharmacopœial directions and a few additional remarks.

ALKALINE CUPRIC TARTRATE VOLUMETRIC SOLUTION

[FEHLING'S SOLUTION]

A. *The Copper Solution.*—Dissolve 34.67 (34.6663) Gm. of carefully selected, small crystals of pure cupric sulphate [*Cupri Sulphas*, U.S. P.], showing no trace of

efflorescence or of adhering moisture, in a sufficient quantity of water to make the solution measure, at 25° C. (77° F.), exactly 500 Cc.

Keep this solution in small, well-stoppered bottles.

B. The Alkaline Tartrate Solution.—Dissolve 173 Gm. of crystallized potassium and sodium tartrate [*Potassii et Sodii Tartras*, U.S. P.] and 75 Gm. of potassium hydroxide [*Potassii Hydroxidum*, U. S. P.] in a sufficient quantity of water to make the solution measure, at 25° C. (77° F.), exactly 500 Cc.

Keep the solution in small, rubber-stoppered bottles.

For use, mix exactly equal volumes of the two solutions at the time required.

One Cubic Centimeter of the mixed solution is the equivalent of:

	Gramme.
Cupric Sulphate, crystallized, $\text{CuSO}_4 + 5\text{H}_2\text{O}$	0.03467
Cupric Tartrate, $\text{CuC}_4\text{H}_4\text{O}_6 + 3\text{H}_2\text{O}$	0.03688
Cane Sugar (Inverted).....	0.00475
Glucose, anhydrous, $\text{C}_6\text{H}_{12}\text{O}_6$	0.00500
Milk Sugar, anhydrous, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$	0.00678

It will be seen from the above that 10 Cc. of mixed solution equals five centigrammes (0.05 Gm.) of glucose. In carrying out the assay, 10 Cc. mixed solution is put into an evaporating dish, is diluted with water (say 50 Cc.), and is brought to the boiling-point.

The diluted sugar solution (usually diabetic urine, as far as the pharmacist is concerned) is put into a burette and is poured into the boiling solution, little by little, until all the copper is precipitated. Details are beyond the scope of this work; hence the reader is referred to one of the books on urinary analysis mentioned in Preface.

The process outlined above involves a peculiar form of calculation; for we see that in this case, contrary to the usual assay, the volumetric solution is measured and the liquid whose strength is to be determined is dropped from the burette. This makes our report read something like this: 10 Cc. Fehling's solution was completely precipitated by 15 Cc. urine. In such case the calculation runs as follows:

10 Cc. Fehling's solution = 15 Cc. urine.
 10 Cc. Fehling's solution = 0.05 Gm. glucose.
 Hence 15 Cc. urine contained 0.05 Gm. glucose.
 Hence 100 Cc. urine contained $\frac{100}{15} \times 0.05$ or 0.333 Gm. glucose.

and we ordinarily say that such urine contains 0.333 per cent. ($\frac{1}{3}$ of 1 per cent.) glucose, although bear in mind that in exact chemical operations percentage should mean grammes to 100 grammes, and not grammes to 100 Cc.

Before leaving Fehling's solution mention should be made of its far more accurate modification—Pavy's solution. This is made by adding 12 Cc. mixed Fehling's solution to 30 Cc. stronger ammonia water, 10 Cc. 10 per cent. soda, and water to make 100 Cc. This mixture is boiled in a flask protected from air by having a layer of melted paraffin floating on its surface. When all the copper is taken up by the urine dropped from the burette, the blue solution becomes colorless. As no precipitate of cuprous oxide forms, the end of the reaction is much easier to note than it is in the Fehling's process.

The above 100 Cc. mixture represents 0.05 Gm. glucose; in short, has exactly the same value as 10 Cc. Fehling's solution.

CHAPTER LVI

GRAVIMETRIC AND GASOMETRIC ASSAYS

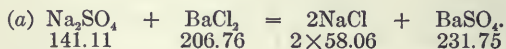
GRAVIMETRIC analysis, as explained on p. 944, is the estimation of the quantity of a certain ingredient of a given substance by the separation of this ingredient, free, in combination, or possibly in reduced form, weighing the solid so separated, and from the weight obtained deducing the amount in the original compound.

Ordinarily, the process consists of precipitating the ingredient as an insoluble compound, but in the final grouping this class of analysis includes such assays as the extracting and weighing of alkaloids (see Opium Assay, p. 1025), the burning of organic salts to carbonates, which are then weighed, and the separation of a button of metal from the ore in the so-called fire assays.

While practically all the pharmacopœial gravimetric assays are either the heating of organic salts to carbonates or strongly heating carbonates to oxides, the assays by precipitation are of such great importance that they merit passing notice.

Precipitation assays are conducted by taking an aqueous solution of a soluble salt, adding thereto the appropriate precipitant (p. 139), collecting the insoluble form of the desired ingredient thus precipitated, and weighing same after carefully washing free from soluble impurity and after heating until all moisture and volatile impurities are driven off. The process can best be explained by a few typical examples.

We want to estimate the amount of sulphuric acid in a certain batch of dried sodium sulphate. We take 0.2 Gm. of the sulphate, dissolve in water, and add barium chloride T.S., when the following reaction occurs:

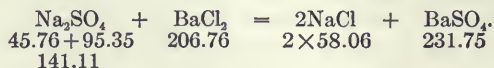


The insoluble barium sulphate is collected on a filter, washed, dried, and weighed, when we find in the case under consideration that 0.2 Gm. Na_2SO_4 gave 0.325 Gm. BaSO_4 .
From the equation above we find:

$$\begin{array}{l} 0.231 \text{ Gm. BaSO}_4 = 0.141 \text{ Gm. absolute Na}_2\text{SO}_4. \\ \text{Then } 0.325 \text{ Gm. BaSO}_4 = \frac{231}{231} \times 0.141 = 0.198 \text{ Gm. absolute Na}_2\text{SO}_4. \end{array}$$

Hence we see that the 0.2 Gm. sodium sulphate contained 0.198 Gm. absolute Na_2SO_4 .

If it is desired to estimate only the acidulous portion (SO_4) of the sodium sulphate, the calculation runs like this:



$$\begin{array}{l} 0.231 \text{ Gm. BaSO}_4 = 0.095 \text{ Gm. SO}_4. \\ \text{Then } 0.325 \text{ Gm. BaSO}_4 = \frac{231}{231} \times 0.095 = 0.133 \text{ Gm. SO}_4. \end{array}$$

We therefore see that

0.2 Gm. Na_2SO_4 contained 0.133 Gm. SO_4 ,
or $\frac{133}{200} \times 100 = 67.5$ per cent. SO_4 .

Another case, showing that the process is applicable to the estimation of the basic constituent of a salt as well as the acidulous portion, might be given. 0.2 Gm. silver nitrate is precipitated by hydrochloric acid as silver chloride, as shown in the following equation:



We find in the assay that 0.2 Gm. silver nitrate gives 0.165 Gm. dried silver chloride.

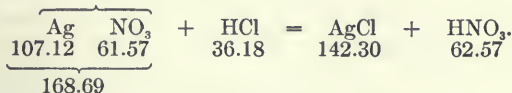
From the equation above we find that—

0.142 Gm. $\text{AgCl} = 0.168$ Gm. absolute AgNO_3 .

Then 0.165 Gm. $\text{AgCl} = \frac{165}{168} \times 0.168 = 0.195$ Gm. absolute AgNO_3 .

Hence 0.200 Gm. of the silver nitrate contained 0.195 Gm. absolute AgNO_3 or $\frac{195}{200} \times 100$ per cent., or 97.5 per cent. absolute AgNO_3 .

Expressed as quantity of metallic silver, the calculation runs like this:



0.142 Gm. $\text{AgCl} = 0.107$ Gm. metallic silver.

Then 0.165 Gm. $\text{AgCl} = \frac{165}{142} \times 107 = 0.124$ Gm. metallic silver.

Hence 0.200 Gm. of the silver nitrate contains 0.124 Gm. metallic silver, or $\frac{124}{200} \times 100$ per cent. = 62 per cent. metallic silver.

The above sketchy outline gives but a glimpse of the important and difficult branch, gravimetric analysis, and for details of the work the reader is referred to the works on the subject mentioned in Preface. Before dismissing the subject the statement should be made that in gravimetric calculations exact molecular weights should be used; that weights and calculations should be carried to the fourth decimal place, and that because of the extreme skill required to obtain accurate results, the process is not used in pharmacopoeial assays.

OFFICIAL GRAVIMETRIC ASSAYS

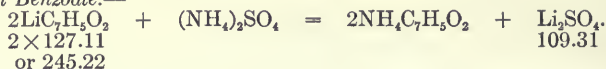
Lithium Benzoate.—If 0.5 Gm. of Lithium Benzoate be thoroughly mixed with about 1 Gm. of powdered anhydrous ammonium sulphate and cautiously ignited in a porcelain crucible, until of constant weight, the residue should weigh not less than 0.210 Gm., nor more than 0.216 Gm.

Lithium Citrate.—See p. 481. 0.5 Gm. salt on ignition with sulphuric acid yields 0.387 to 0.394 Gm. residue.

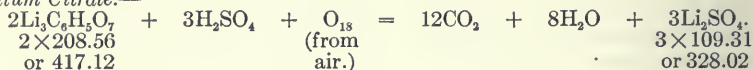
Lithium Salicylate.—Like assay for lithium benzoate, except residue must weigh between 188 and 192 milligrammes.

The residue in each of these cases is lithium sulphate, so we see that the three salts yield respectively 42 per cent., 77 per cent., and 37 per cent. lithium sulphate. The reaction whereby the sulphate is formed in each case is given below:

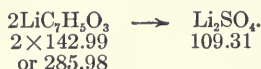
Lithium Benzoate.—



Lithium Citrate.—



Lithium Salicylate.—



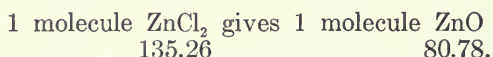
By dividing the molecular proportion of lithium sulphate, shown in above equations, by the molecular proportions of the special salt, the percentages given above will be obtained.

Precipitated Zinc Carbonate.—One Gm. of Zinc Carbonate, after strong ignition in a porcelain crucible, should leave a residue of zinc oxide weighing not less than 0.720 Gm.

The percentage of zinc oxide yielded by official zinc carbonate (72 per cent.) cannot be well calculated, since the composition of zinc carbonate, as explained on p. 522, is variable.

Zinc Chloride.—Dissolve 0.5 Gm. of Zinc Chloride in 200 Cc. of boiling distilled water, add about 5 drops of phenolphthalein T.S., and then sufficient sodium carbonate T.S., with constant stirring, to cause the solution to assume a permanent red color; transfer the resulting precipitate to a plain filter, wash it with boiling distilled water until all soluble matter has been removed, then dissolve it in a sufficient quantity of nitric acid, evaporate the sodium to dryness, and ignite the salt until it ceases to lose weight; the residue should weigh not less than 0.297 Gm.

This assay means the conversion of zinc chloride into zinc oxide, and is based on exact molecular proportions, viz:



$$\frac{.50}{135.26} \times 80.78 = 0.298.$$

Hence 0.500 Gm. zinc chloride gives 0.298 Gm. zinc oxide.

Gold and Sodium Chloride.—See p. 586.

On account of the variability of this salt, the 30 per cent. limit of metallic gold does not represent the exact theoretic amount, which would be about 32 per cent.

Bismuth Subnitrate.—If 2 Gm. of the salt be heated in a porcelain crucible until nitrous vapors cease to be evolved, the residue of bismuth oxide, when cold, should weigh not less than 1.6 Gm.

Bismuth Subcarbonate.—If 1 Gm. of Bismuth Subcarbonate be thoroughly ignited at red heat in a porcelain crucible, the residue of bismuth oxide should weigh not less than 0.9 Gm.

Bismuth Citrate.—If 1 Gm. of Bismuth Citrate be thoroughly ignited in a porcelain crucible, and, after cooling, 5 Cc. of nitric acid be added to the residue, drop by drop, warming until complete solution is effected, then evaporating to dryness, and again igniting, a residue of bismuth oxide should be left weighing not less than 0.58 Gm. nor more than 0.6 Gm.

Bismuth and Ammonium Citrate.—Like assay of bismuth citrate, except yield of bismuth oxide is not less than 0.48 Gm.

Bismuth Subgallate.—Like assay of bismuth citrate, except yield of bismuth oxide is 0.52 to 0.57 Gm.

Bismuth Subsaliicylate.—Like assay of bismuth citrate, except yield of bismuth oxide is 0.62 to 0.64 Gm.

Comparing the above bismuth assays we find:

Two Gm. bismuth subnitrate should yield 1.6 Gm. bismuth oxide—80 per cent.

One Gm. bismuth subcarbonate should yield 0.9 Gm. bismuth oxide—90 per cent.

One Gm. bismuth citrate should yield about 0.58 Gm. bismuth oxide—58 per cent.

One Gm. bismuth and ammonium citrate should yield 0.48 Gm. bismuth oxide—48 per cent.

One Gm. bismuth subgallate should yield 0.52 Gm. bismuth oxide—52 per cent.

One Gm. bismuth subsaliicylate should yield 0.62 Gm. bismuth oxide—62 per cent.

Since all these bismuth salts are of variable composition, the calculations of these percentages by molecular proportions are not easy.

There are two gravimetric assays in which alkaloids are extracted from salts by the "shaking-out" process and then weighed.

Iron and Quinine Citrate.—*Assay for the Quinine.*—Introduce 1.11 Gm. of Iron and Quinine Citrate into a dish, and, with the aid of a gentle heat, dissolve it in 20 Cc. of water. Transfer the solution, together with the rinsings of the dish, to a separator, allow the liquid to become cold, then add 5 Cc. of ammonia water and 10 Cc. of chloroform, and shake the separator for one minute. Allow the liquids to separate, draw off the chloroformic layer and shake the residuary liquid a second and a third time with portions of 10 Cc. each of chloroform. Allow the combined chloroformic solutions to evaporate spontaneously in a tared dish, and dry the residue at 100° C. (212° F.) to a constant weight. This residue should weigh not less than 0.1726 Gm. (corresponding to at least 11.5 per cent. of dried quinine), and should conform to the reactions and tests under *Quinina*.

Iron and Strychnine Citrate.—*Assay for the Strychnine.*—Dissolve 4.44 Gm. of Iron and Strychnine Citrate, in a separator, in 15 Cc. of water, add 5 Cc. of ammonia water and 10 Cc. of chloroform, and shake the separator for one minute. Allow the liquids to separate, draw off the chloroformic layer, and shake the residuary liquid a second and a third time with portions of 10 Cc. each of chloroform. Allow the combined chloroformic liquids to evaporate spontaneously in a tared dish, and dry the residue at 100° C. (212° F.) to a constant weight. This residue should weigh not less than 0.04 (0.0399) Gm., nor more than 0.0444 Gm. (corresponding to not less than 0.9 nor more than 1 per cent. of strychnine), and should respond to the reactions and tests given under *Strychnina*.

GASOMETRIC ANALYSIS

This form of analysis is performed by liberating certain gases from appropriate substances by chemical action, measuring amount of gas obtained in a nitrometer (Fig. 232) or other measuring tube for gases; from the volume of the gas calculate its weight, and from its weight calculate the amount of chemical from which the gas was liberated. Only two official chemicals are assayed by this process,—spirit of

nitrous ether and amyl nitrite,—and as the principle of assay was explained in the discussion of the preparation first mentioned (p. 662), it is unnecessary at this place to do more than insert the pharmacopœial description of the process.

GASOMETRIC ESTIMATION

In certain cases the Pharmacopœia directs the strength of a product or chemical substance to be determined by the volume of gas (nitrogen dioxide) given off during a definite reaction. This volume is to be determined by the nitrometer in the following manner:

Arrange a nitrometer consisting of a measuring tube (graduated for at least 50 Cc.) connected by stout rubber tubing with an open equilibrium tube (both tubes, preferably, provided with a globular expansion near the lower end) in such a manner, by suitable clamps attached to a stand, that either tube may be readily and quickly clamped at a higher or lower level. The stop-cock of the measuring tube having been opened, and the open equilibrium tube having been raised to a higher level, pour into the latter a saturated aqueous solution of sodium chloride, until the measuring tube, including the bore of the stop-cock, is completely filled. Then close the latter and fix the equilibrium tube at a low level. Having ascertained that the stop-cock is closed air-tight, and having, if necessary, wiped out the graduated tube of the nitrometer, introduce into it the prescribed quantity of the liquid to be tested, and allow this to flow slowly into the measuring tube, being careful not to admit any air. Follow it by the prescribed quantities of the several reagents (potassium iodide T.S. and normal sulphuric acid V.S.). When the reaction, which takes place at once, moderates, remove the measuring tube from its clamp, and, being careful to hold it constantly so that the liquid contained in it stands at a higher level than that in the equilibrium tube, shake its contents, without permitting any gas to pass into the open tube. When the reaction has completely ceased, restore the tube to its fastening, and allow the apparatus and contents to acquire the ordinary temperature of the room, which is assumed to be at or about 25° C. (77° F.). Then adjust the two tubes so that the liquid columns are at exactly the same level, and read off the volume of gas in the measuring tube. Multiply this figure by the weight of the substance, which is the equivalent of 1 Cc. of nitrogen dioxide. The result will be the weight of the pure substance (nitrite) contained in the amount taken for the assay.

For pharmacopœial purposes the determination will be sufficiently exact if the evolved gas be measured at or near 25° C. (77° F.).

Since temperature and barometric pressure materially affect the volume of the gas, the following correction factors must be used to obtain reasonably exact results when the temperature and pressure are not nearly normal. The barometer correction is important at any locality more than 250 meters above sea-level:

FACTORS FOR TEMPERATURE CORRECTIONS

(Normal Temperature, 25° C.)

Temperature.	Factor.	Temperature.	Factor.	Temperature.	Factor.
15° C.	1.035	22° C.	1.010	29° C.	0.987
16° C.	1.031	23° C.	1.007	30° C.	0.983
17° C.	1.028	24° C.	1.003	31° C.	0.980
18° C.	1.024	25° C.	1.000	32° C.	0.977
19° C.	1.021	26° C.	0.997	33° C.	0.974
20° C.	1.017	27° C.	0.993	34° C.	0.971
21° C.	1.014	28° C.	0.990	35° C.	0.968

EXAMPLE.—Assuming that the volume of gas read off was 44.5 Cc. at 32° C. (89.6° F.), and it is desired to ascertain the corresponding volume at 25° C. (77° F.), barometric pressure not being taken into consideration, then the 44.5 Cc. must be reduced in the proportion of 1 to 0.977 (see temperature correction factors above), or 44.5 must be multiplied by 0.977. The result will be 43.48 (43.4765) Cc. as the equivalent volume of gas at 25° C. (77° F.).

FACTORS FOR CORRECTION FOR BAROMETRIC PRESSURE

(Normal Barometer, 760 Mm.)

Barometer Reading.		Factor.	Barometer Reading.		Factor.
Mm.	Inches.		Mm.	Inches.	
790	31.10	1.039	660	25.98	0.868
780	30.71	1.026	650	25.59	0.855
770	30.31	1.013	640	25.20	0.842
760	29.92	1.000	630	24.80	0.829
750	29.53	0.987	620	24.41	0.816
740	29.13	0.974	610	24.02	0.803
730	28.74	0.961	600	23.62	0.789
720	28.35	0.947	590	23.23	0.776
710	27.95	0.934	580	22.83	0.763
700	27.56	0.921	570	22.44	0.750
690	27.17	0.907	560	22.05	0.737
680	26.77	0.895	550	21.65	0.724
670	26.38	0.882			

EXAMPLE.—Assuming that the volume of gas read off was 43.48 (43.4675) Cc. at 590 Mm. barometric pressure, and it is desired to ascertain the corresponding volume at normal barometric pressure (760 Mm.), temperature not being taken into consideration, then the 43.48 Cc. must be reduced in the proportion of 1 to 0.776 (see barometric correction factors above), or 43.48 must be multiplied by 0.776. The result will be 33.74 Cc. as the equivalent volume of gas at normal barometric pressure.

ESTIMATION OF NITROGEN DIOXIDE

NO=29.81; 1 Liter $\left\{ \begin{array}{l} \text{at } 0^{\circ} \text{ C. and 760 Mm.} = 1.3396 \text{ Gm.} \\ \text{at } 25^{\circ} \text{ C. and 760 Mm.} = 1.2272 \text{ Gm.} \end{array} \right.$

One Cubic Centimeter of Nitrogen Dioxide is the equivalent of:

	At 0° C. and 760 Mm. Gramme.	At 25° C. and 760 Mm. Gramme.
Nitrogen Dioxide, NO=29.81.....	0.0013396	0.0012272
Amyl Nitrite, C ₅ H ₁₁ NO ₂ =116.24.....	0.0052234	0.0047851
Ethyl Nitrite, C ₂ H ₅ NO ₂ =74.51.....	0.0033482	0.0030673
Sodium Nitrite, NaNO ₂ =68.57.....	0.0030813	0.0028227

PART V.

CHAPTER LVII

THE PRESCRIPTION

HAVING covered the entire field of special pharmaceutic operations and preparations, we can proceed to dispensing pharmacy and to that climax of the pharmaceutic art, the dispensing of prescriptions. Very properly, the prescription is the last topic studied in a course on pharmacy, for the preparation of prescriptions calls into play all the knowledge that belongs to an educated pharmacist. Not only must the prescriptionist know pharmaceutic manipulations and be well acquainted with all pharmaceutic preparations, but he must, moreover, be thoroughly familiar with all possibilities of chemical reaction, with the constituents of crude drugs, and, of course, must be well up on posology—dosage. However, we will leave the important topic of prescription criticism for subsequent pages, and this chapter will be devoted to the generalities of dispensing and to a discussion of the general character of the prescription—its composition, translating, and dispensing.

The *prescription* is an order written by the physician calling for medicines to be compounded and dispensed by the pharmacist.

Technically, the ideal prescription is supposedly divided into several parts, which can best be explained after presentation of a typical prescription:

Ideal Prescription.

(Date.)		August 6, 1908.
(Name of Patient.)	FOR MRS. JONES.	
(Superscription.)	R _x .	
(Inscription.)	{ Acidi salicylici..... Potassii acetatis..... Glycerini..... Aquæ.....q. s. ad.	{ ℥iij. (Basis.) ℥iv. (Adjuvant). ℥j. (Corrective.) ℥iv. (Vehicle.)
(Subscription.)	Misce fiat solutio.	
(Signa.)	Sig.—One teaspoonful four times a day.	
(Name of doctor.)		DR. BROWN.

Taking up the parts of the prescription in regular order, the following information may be of service:

The *date* of a prescription is rarely written by the physician, but is of value in establishing its identity.

The *name of the patient* should always be written by the physician, and especially the information as to whether the patient is an infant, child, or adult. If the doctor omits this important datum, the pharmacist should supply it by writing the name and address on the prescription or on the card to which it is attached. There is usually little difficulty in persuading the patient to furnish name and address. To this may be added the emphatic statement that whenever the

name does appear, it should be written on the label. Suppose, in handing the medicine to two customers, an exchange is effected, and Mrs. Smith gets the remedy intended for Mrs. Jones. If the label bears the inscription "For Mrs. Jones," that inscription might prove a warning and secure the return of the medicine to the pharmacy. Of course, such an error on the part of the pharmacist is scarcely excusable, but such errors sometimes occur, and lucky is the man who is saved from the possible disastrous results of a slip of that kind by the simple precaution just given.

The *superscription* is the sign which is an abbreviation of the Latin word "recipio," meaning "take thou." The flourish at the end of the R is presumably the sign of Jupiter, legend saying that Roman physicians used the sign, thus making the superscription an invocation "take thou in the name of Jupiter." This pleasant fable has never, however, been well authenticated. The French use the abbreviation "P," meaning "prenez," "take."

The *inscription* is the body of the prescription—the enumeration of the medicines desired and the quantity of each. As seen above, the inscription of the typical prescription may contain four groups of medicinal agents, although the modern physician, is rapidly abandoning complex prescriptions, often prescribing a single ingredient.

Explaining these four groups:

The *basis* is the main ingredient—the medicine first indicated to the mind. Thus, in the above prescription (evidently for a rheumatic patient) the remedy first suggested was salicylic acid.

The *adjuvant* is the medicine intended to aid the basis in its medical action. In the above prescription the doctor evidently recalled that rheumatism is due to excess of uric acid, and decided to attempt to remove the uric acid by the use of the diuretic, potassium acetate. Hence this salt is the adjuvant—the assistant.

The *corrective* is some substance added to lessen irritating effect or improve flavor of the main ingredients. In the prescription given above glycerin is the corrective, but frequently flavored syrups or non-medicated elixirs are used as correctives.

The *vehicle*, or *diluent*, is the material added to dilute the preparation to a reasonable dose. Too much stress cannot be laid upon the danger of prescribing small doses, such as a few drops of a liquid. In the hands of the ignorant or careless fatal mistakes are apt to occur.

Most dangerous of all is the custom of some physicians in directing the patient to dilute a potent prescription at home. A fatal case of this kind occurring in Cleveland was the prescribing of tincture of aconite (U. S. P. 1890) for an infant, with the following remarkable directions: "Five drops in a glass of water, and then a teaspoonful every hour." The mother followed the directions implicitly, giving the babe five drops of the tincture in water, followed by a teaspoonful of the potent tincture!

The *subscription* includes the directions as to compounding intended for the guidance of the pharmacist. In the olden times the subscription was frequently quite lengthy, minute details of the compounding being written in choice Latin, but at present the subscription is usually boiled down to the succinct "M" or "M ft. pil." or "F. S. A." These terms are left untranslated, that the student may hunt up the abbreviations in the list of Latin terms found on a subsequent page.

PRINCIPLES OF PHARMACY

signa represents the directions to the patient, which directions are to be written on the label by the pharmacist, translating from Latin to English, if the physician gives the directions in the former language. Like the subscription, the *signa* was formerly written by the physician in Latin, but the modern medical man, not being as much at home with the classics as were his predecessors, rarely resorts to Latin, except a few time-worn abbreviations found in the list of Latin terms.

The modern physician, alas! pays but little attention to his prescription, and there is no part of the curriculum of medical colleges more neglected than the very important branch of prescription writing. It is sadly true that one reason why so many young physicians have gotten into the pernicious practice of prescribing ready-made nostrums is because of their lack of knowledge of correct prescription writing.

The pharmacist should do all in his power to dissuade the physician from reducing his "*signa*" to the stereotyped "as directed," since the dosage should appear on the label for the guidance of the patient, and on the prescription that the pharmacist may determine if the dose is safe. While on the question of dosage, mention should be made of the need of adoption, in this country, of the system regulating large doses that is in vogue in Germany. In that country an authoritative list of maximum doses is published, and any physician desiring to prescribe a dose larger than the maximum declares his intention by underscoring the dose, or rewriting the quantity in words.

As shown above, the ideal prescription is written in the Latin language, which is used because it is the universal language of science; because it lessens the use of local synonyms; and because it is frequently advisable to keep the patient from knowing the nature of the medicine prescribed.

Concerning the value of Latin as the universal language of science, suppose an actual case and imagine we have before us three prescriptions—one of an American practitioner, one written by a physician of Hanover, Germany, and the last the order of a doctor practising in Buda-Pest, Hungary. Quite likely the pharmacist knows nothing of German, almost certainly he is not acquainted with the weird Hungarian tongue, yet were all three prescriptions brought to his store, he could read one as easily as the other, for all are written in Latin.

The other great value of the use of Latin is that it practically does away with the use of confusing synonyms. A person comes into a drug-store and asks for "*chamomile*," and the pharmacist has to inquire "*which chamomile—German or Roman.*" Even so would it be if the doctor wrote his prescription in English and demanded "*extract of chamomile.*" The question would again be, "*which chamomile?*" while if he writes "*ext. anthemidis,*" or "*ext. matricariæ,*" the chamomile he wants is very plain.

PRESCRIPTION LATIN

In the brief space of one chapter, much idea of Latin cannot be given, but the following explanations may throw at least some light on the Latin of prescriptions.

In most English grammars we find that nouns are said to have three "cases"—nominative, possessive, and objective. Latin nouns have six such cases, but of these only two are used to any extent in pharmacy—the nominative and the genitive.

Those who have studied English grammar at all, will remember that the nominative case means that form of noun or pronoun used as the subject of the verb; thus, "John" is nominative in the sentence "John runs"; likewise "I" in the sentence "I walk." In English the form of the noun is the same, regardless of its case. Not so, however, with pronouns, for while "I" is nominative, "me" is its objective form. Latin nouns have a different ending for each case, just as English pronouns differ according to case. Having thus described the nominative, we can turn to the genitive case, which has no exact counterpart in English. It comes nearest to our possessive case, but it really means what we would put in English as "of the—," supplying the noun for the dash. A grouping of the terminations of the Latin noun in its various cases is called its declension, and the Latin nouns are grouped into five declensions, each having a different set of endings. Thus, in the first declension, we have—

Nominative, mensa (a table).

Genitive, mensæ (of a table).

and the plural of the same noun is

Nominative, mensæ (tables).

Genitive, mensarum (of tables).

In our limited space we cannot go beyond these few facts, and we will immediately proceed to practise the two cases as found in the Latin names of the pharmacopœia:

First, let us look up a pharmacopœial name belonging to the declension just given—the first. This we find in two words, among many others, "tinctura" and "serpentaria."

Nominative, tinctura (a tincture); serpentaria (serpentaria).

Genitive, tincturæ (of a tincture); serpentariæ (of serpentaria).

Let us bring these together in the official name, tincture of serpentaria, which in Latin is tinctura serpentariæ.

In the second declension we find two forms of nouns—masculine nouns ending in "us", and neuter nouns ending in "um." They are declined:

Nominative, us um

Genitive, i i

Let us apply these terminations, first picking out some official noun ending in "us"; for instance, "syrupus" and "rubus":

Nominative, syrupus (syrup); rubus (blackberry).

Genitive, syrupi (of syrup); rubi (of blackberry).

Combining these to read "syrup of blackberry," we get "syrupus rubi."

Among the official nouns ending in "um," we find "unguentum" and "iodum."

Nominative, unguentum (ointment); iodium (iodine).

Genitive, unguenti (of ointment); iodi (of iodine).

Then the official name of "ointment of iodine" is "unguentum iodi." Of course, we can combine nouns of any declension, only

being careful to employ the correct termination. Thus, we can write "tinctura iodi" (tincture of iodine) or "syrupus serpentariæ" (syrup of serpentaria).

The third declension embraces all those nouns whose genitive ends in "is." The nominative varies greatly, causing the declension to be complex, but all the pharmacopœial variations will be given here:

<i>Nominative.</i>	<i>Genitive.</i>
Adeps (lard).	Adipis (of lard).
Macis (mace).	Macis (of mace).
Sulphas (a sulphate).	Sulphatis (of a sulphate).
Sulphis (a sulphite).	Sulphitis (of a sulphite).
Mucilago (a mucilage).	Mucilaginis (of a mucilage).
Nux (a nut).	Nucis (of a nut).
Liquor (a solution).	Liquoris (of a solution).
Rhus (rhus).	Rhois (of rhus).
Flos (a flower).	Floris (of a flower).
Theobroma (theobroma).	Theobromatis (of theobroma).
Cantharis (cantharides).	Cantharidis (of cantharides).

In "rhus" we have a noun ending in "us" which does not belong to the second declension, and likewise the entire fourth declension ends in "us." Fortunately, there are but two pharmacopœial names involved in this declension; they being "spiritus" and "fructus," which are declined as follows:

<i>Nominative,</i> spiritus (a spirit).	<i>Genitive,</i> spiritus (of a spirit).
" fructus (a fruit).	" fructus (of a fruit).

To sum up, all pharmacopœial nouns ending in "um" take the termination "i" in the genitive; those in "a" in the nominative—except theobroma—take "æ" in the genitive; and all ending "us" in the nominative—except rhus, fructus, and spiritus—take "i" in the genitive; these rules applying only to the singular number.

Concerning the plural, we need only consider that of the first, second, and third declensions, exemplified in "herba," "folium," and "flos" respectively.

SINGULAR.		PLEURAL.	
<i>Nominative.</i>	<i>Genitive.</i>	<i>Nominative.</i>	<i>Genitive.</i>
Herba (an herb).	Herbæ (of an herb).	Herbæ (herbs).	Herbarum (of herbs).
Folium (a leaf).	Folii (of a leaf).	Folia (leaves).	Foliorum (of leaves).
Flos (a flower).	Floris (of a flower).	Flores (flowers).	Florum (of flowers).

So far we have considered the subject only in the simple relationship as seen in pharmacopœial names, and to make the matter still more clear, we will cite a few names where the application of the declensions may be seen:

Quininae sulphas,	Sulphate of quinine.
Sodii sulphis,	Sulphite of sodium.
Mucilago Acaciæ,	Mucilage of gum arabica.
Tinctura nucis vomicæ,	Tincture of nux vomica.
Liquor calcis,	Solution of lime.
Fluidextractum rhois glabræ,	Fluidextract of rhus glabra.
Spiritus ætheris,	Spirit of ether.
Tinctura belladonnæ foliorum,	Tincture of belladonna leaves.
Tinctura arnicæ florum,	Tincture of arnica flowers.
Tincturæ herbarum recentium,	Tinctures of fresh herbs.

("Recentium" is an adjective, genitive plural, from the nominative singular "recens," and genitive singular "recentis.")

The pharmacopœial name (or the Latin nominative singular) of "nux vomica," "rhus glabra," and "sodium" are the words just given.

We are now ready to consider the changes the Latin name undergoes in writing the prescription—a change that is quite marked, though very simple.

The sign *R* means "take" and nothing else, and in the prescription we are not supposed to write—

"Take, sulphate of quinine," but, "Take, *of* sulphate of quinine," which means that in translating the prescription into Latin the word "sulphate" is put into the genitive, the same as is the word "quinine," and when correctly written, the term will stand—

"*R. Quininæ sulphatis*"

Take *of* sulphate *of* quinine (reversed, you see) and *not*—

"*R. Quininæ sulphas*"

Take sulphate *of* quinine.

This is the only change in writing the pharmacopœial name in a prescription, although sometimes this change is perplexing; thus we write:

R. Mucilaginis acaciæ and *not R. Mucilago acaciæ*
(*of* mucilage *of* acacia) (mucilage *of* acacia).

Besides the nominative and genitive cases, the *ablative* case is used occasionally in pharmacopœial titles. Even as the genitive is used after the preposition "*of*," so the ablative is used after the prepositions "*from*," "*with*," "*in*," and "*by*." The following are the pharmacopœial instances of the ablative:

Hydrargyrum cum creta—mercury with chalk. In this case "*creta*" is the ablative singular of noun of the first declension, *creta*.

Emulsum olei morrhuæ cum hypophosphitibus—emulsion of cod liver oil with hypophosphites. In this case "*hypophosphitibus*" is the ablative plural of the noun of the third declension, *hypophosphis*.

Ferri hydroxidum cum magnesiæ oxido—hydrated oxide of iron with oxide of magnesium. In this case "*oxido*" is the ablative singular of the noun of the second declension, *oxidum*.

The above outline refers only to Latin nouns. *Adjectives* change number and case with the noun they qualify, but their terminations do not necessarily conform with the termination of the noun. Thus, a masculine noun of the third declension may be qualified by a masculine adjective of the second declension. It simply changes to the same gender and case. Therefore it might be well to insert a list of adjectives found in pharmacopœial titles with their declensions:

FIRST AND SECOND DECLENSIONS.

Singular.

	<i>Masculine.</i>	<i>Feminine.</i>	<i>Neuter.</i>
<i>Nominative</i> —	<i>adhesivus</i>	<i>adhesiva</i>	<i>adhesivum</i>
<i>Genitive</i> —	<i>adhesivi</i>	<i>adhesivæ</i>	<i>adhesivi</i>

Plural.

	<i>Masculine.</i>	<i>Feminine.</i>	<i>Neuter.</i>
<i>Nominative</i> —	<i>adhesivi</i>	<i>adhesivæ</i>	<i>adhesiva</i>
<i>Genitive</i> —	<i>adhesivorum</i>	<i>adhesivarum</i>	<i>adhesivorum.</i>

Adjectives of this class found in pharmacopœial titles are:

Adhesivus	in emplastrum adhesivum	(adhesive plaster).
Amarus	in aurantium amarum	(bitter orange).
	in amygdala amara	(bitter almond).
	in vinum ferri amarum	(bitter wine of iron).
Ammoniatus	Tinctura guaiaci ammoniata	(ammoniated tincture of guaiac).
Benzoinatus	in adeps benzoïnatus	(benzoïnated lard).
Camphoratus	in tinctura opii camphorata	(camphorated tincture of opium).
Cantharidatus	in collodium cantharidum	(cantharidal collodion).
Citratus	in caffeina citrata	(citrated caffeine).
Compositus	Tinctura cinchonæ composita and a number of others.	(compound tincture of cinchona).
Corrosivus	in hydrargyri chloridum corrosivum.	(corrosive chloride of mercury).
Deodoratus	in tinctura opii deodorata	(deodorized tincture of opium).
Despumatus	in mel despumatum	(clarified honey).
Destillatus	in aqua destillata	(distilled water).
Dilutus	in acidum sulphuricum dilutum and a number of others	(diluted sulphuric acid).
Expressus	in oleum amygdalæ expressum	(expressed oil of almond).
Exsiccatus	in alumen exsiccatum	(exsiccated alum).
	in ferri sulphas exsiccatus	(exsiccated sulphate of iron).
	in sodii phosphas exsiccatus	(exsiccated phosphate of sodium).
Flavus	in hydrargyri oxidum flavum	(yellow oxide of mercury).
Fusus	in argenti nitras fusus	(fused nitrate of silver).
Granulatus	in ferri sulphas granulatus	(granulated sulphate of iron).
Lotus	in sulphur lotum	(washed sulphur).
Mitigatus	in argenti nitras mitigatus	(mitigated nitrate of silver).
Monohydratus	in sodii carbonas monohydratus	(monohydrated carbonate of sodium).
Ponderosus	in magnesiï oxidum ponderosum.	(heavy oxide of magnesium).
Præcipitatus	in ferri sulphas præcipitatus and others.	(precipitated sulphate of iron).
Reductus	in ferrum reductum	(reduced iron).
Saccharatus	in ferri carbonas saccharatus	(saccharated carbonate of iron).
Stypticus	in collodium stypticum	(styptic collodion).
Sublimatus	in sulphur sublimatum	(sublimed sulphur).
Sulphuratus	in calx sulphurata	(sulphurated lime).

The adjective *ruber* (example hydrargyri oxidum rubrum—red oxide of mercury) has same endings in the nominative and genitive cases as the other adjectives of the first and second declensions, except that the masculine nominative singular is *ruber*.

THIRD DECLENSION OF ADJECTIVES.

	<i>Singular.</i>		<i>Plural.</i>
	<i>Masculine and Feminine.</i>	<i>Neuter.</i>	<i>Masculine and Feminine.</i>
<i>Nominative—</i>	Animalis	animale	animales
<i>Genitive—</i>	Animalis	animalis	animalium
			<i>Neuter.</i>
			animalia
			animalium.

Adjectives of this class found in pharmacopœial titles are:

Animalis	in carbo animalis	(animal charcoal).
Dulcis	in amygdala dulcis	(sweet almond).
Flexilis	in collodium flexile	(flexible collodion).
Glacialis	in acidum aceticum glaciale	(glacial acetic acid).
Levis	in magnesiï oxidum levis	(light oxide of magnesium).
Mitis	in hydrargyri chloridum mite	(mild chloride of mercury).
Mollis	in sapo mollis	(soft soap).
Solubilis	in ferri phosphas solubilis and several others.	(soluble phosphate of iron).

Some adjectives of the third declension have a slightly different form, viz.:

<i>Singular.</i>			<i>Plural.</i>	
<i>Masculine and Feminine.</i>	<i>Neuter.</i>		<i>Masculine and Feminine.</i>	<i>Neuter.</i>
<i>Nominative</i> —adjuvans	adjuvans		adjuvantis	adjuvantia
<i>Genitive</i> —adjuvantis	adjuvantis		adjuvantium	adjuvantium.

Three of these are used in pharmacopœial titles:

Adjuvans	in elixir adjuvans	(adjuvant elixir).
Effervescens	in pulvis effervescens compositus	(compound effervescent powder).
Recens	in tincturæ herbarum recentium	(tinctures of fresh herbs).

Three pharmacopœial titles have the adjective in the comparative degree:

Aqua ammoniæ fortior	(stronger ammonia water).
Aqua aurantii florum fortior	(stronger orange flower water).
Aqua rosæ fortior	(stronger rose water).

Verbs are not used much in modern prescription writing beyond the stereotyped abbreviated phrases found in the list below. Moreover, a discussion of Latin verbs and their conjugation would mean a complete review of Latin grammar, which is clearly beyond the scope of this work. For further details, therefore, the reader is referred to list of books on pharmaceutic Latin found in Preface.

TABLE OF LATIN AND FOREIGN TERMS USED IN PRESCRIPTIONS

[(L.) Latin, (G.) German, (F.) French.]

TERM.	ABBREVIATION.	MEANING.
Abendessen (G.),		Supper.
Ad (L.),		To, up to.
Addē (L.),	Add.	Let it be added.
Ad libitum (L.),	Ad lib.	At pleasure.
Alternis horis (L.),		Every other hour.
Ana (L.),	āā.	Of each.
Ante (L.),		Before.
Aqua bulliens (L.),	Aq. bull.	Boiling water.
Aqua fervens (L.),	Aq. ferv.	Warm water.
Aqua fontis (L.),	Aq. font.	Spring water.
Aqua pluvialis (L.),	Aq. pluv.	Rain water.
Alle drei stunden (G.),		Every three hours.
Bene (L.),		Well, good.
Bis in die (L.),	B. i. d.	Twice a day.
Bissen (G.),		Bolus.
Bol (F.),		Bolus.
Capia (L.),	Cap.	Let the patient take.
Capsulæ amylicæ (L.),		Starch capsules (cachets).
Charta cerata (L.),	Chart, cer.	Waxed paper.
Chartula (L.),	Cht.	Papers.
Cochleare amplum (L.),	Coch. ampl. }	Tablespoonful.
Cochleare magnum (L.),	Coch. mag. }	
Cochleare medium (L.),	Coch. med.	Dessertspoonful.
Cochleare parvum (L.),	Coch. parv.	Teaspoonful.
Coletur (L.),		Let it be strained.
Collyrium (L.),	Collyr.	Eye-wash.
Coquetur (L.),	Coq.	Let it be boiled.

TERM.	ABBREVIATION	MEANING.
Coucher (F.),		Bedtime.
Cuillerée a café (F.),		Teaspoonful.
Cuillerée a soupe (F.),		Tablespoonful.
Detur (L.),	Det. }	Let it be given.
Dentur (L.),	Dent. }	
Dentur talis dosis (L.),	Dent. tal. dos.	Give of such doses.
Deux (F.),		Two.
Dies (L.),	D.	Day.
Dividatur (L.),	Divid.	Let it be divided.
Dosis (L.),		A dose.
Drachma (L.),		A drachm.
Drei mal täglich (G.),		Three times a day.
Ejusdem (L.),		Of the same.
Enemata (L.),	Enem.	Enema.
Esslöffelvoll (G.),		Tablespoonful.
Et (L.)		And.
Ex mode prescripto (L.),	E. m. p.	{ As prescribed.
Fiat (L.),	Ft.	{ As directed.
Fois (F.),		Let it be made.
Gouttes (F.),		Time.
Granum (L.),	Gr.	Drops.
Guttæ (L.),	Gtt.	Grain.
Hora (L.),		Drops.
Hori somni (L.),	Hor. som.	An hour.
Jeder or jeden (G.),		At bedtime.
Latwerge (G.),		Every.
Mica panis (L.),	Mic. pan.	Electuary.
Misce (L.),	M.	Crumb of bread.
Mittagessen (G.),		Mix.
Mitte talis dosis (L.),		Dinner (at noon).
Non repetatur (L.),	Non. rep.	Send of such doses.
Numero (L.),	No.	Do not repeat.
Oblate (G.),		In number.
Omni horæ (L.),	Omn. hor.	Wafer paper.
Omni mane (L.),	Omn. man.	Every hour.
Omni nocte (L.),	Omn. noct.	Every morning.
Optimus (L.),	Opt.	Every night.
Pain azyme (F.),		The best.
Paquet (F.),		Wafer paper.
Partes æqualis (L.),	P. æ.	A powder (package).
Pastillen (G.),		Equal parts.
Pillen (G.) }		Lozenges.
Pilule }		Pills.
Pondus civile (L.),		Avoirdupois weight.
Pondus medicinale (L.),		Apothecary weight.
Post cibo (L.),	Post. cib.	After eating.
Poudre (F.),		Powder.
Pro re nata (L.),	P. r. n.	As occasion requires (when needed).
Pulver (G.),		Powder.
Pulvis (L.),	Pulv.	A powder.
Pulvis subtilissimus (L.),	Pulv. subtil.	Very smooth powder.
Quantum sufficit (L.),	Q. s.	Sufficient quantity.
Quotidie (L.),		Daily.
Repetatur (L.),	Rept.	Let it be repeated.
Rotula (L.),		Globules.
Schlafengehen (G.),		Bedtime.
Scrupulus (L.),	℥	A scruple.
Secundum artem (L.),	S. a.	According to art.
Sesqui (L.),		Once and a half.
Signetur (L.),	Sig.	Let it be marked.
Solvetur (L.),	Solve.	Let it be dissolved.
Spiritus Vini Rectificata (L.),	S. V. R.	Alcohol.

TERM.	ABBREVIATION.	MEANING.
Spiritus Vini Tenuis (L.),	S. V. T.	Diluted Alcohol.
Tabella (L.),	Tab.	A lozenge.
Talis (L.)		Of such.
Ter (L.),		Thrice.
Ter in die (L.),	T. i. d.	Thrice daily.
Tere bene (L.),		Rub well.
Theelöffelvoll (G.),		Teaspoonful.
Toutes les heures (F.),		All the hours (every hour).
Trois fois par jour (F.),		Three times a day.
Tropfen (G.),		Drops.
Uncia (L.),	℥	An ounce.
Ut dictum (L.),	Ut. dict.	As directed.
Zwei (G.),		Two.

For a more complete list of Latin terms, see Scoville's "Art of Compounding."

While on the subject of abbreviations, mention should be made of the danger likely to arise from the careless writing of abbreviated names of drugs. Among classic instances of such abbreviations are "hyd. chlor," which might mean chloral hydrate, mercuric chloride, or mercurous chloride; "liq. mag," which might mean solution of some magnesium salt, or else Magendie's solution (p. 910), while "sulph." may mean sulphate, sulphite, or sulphide; "nit." may mean nitrate or nitrite, and similarly an abbreviation of name of any negative element in salts and acids can be misconstrued when there is more than one acid derived from the element. In most cases of such abbreviation the experienced pharmacist can discern the remedy desired either by the dose prescribed or by the relative popularity of the several possibilities. Thus, a prescription calling for "zinc sulph." almost invariably means zinc sulphate, as the sulphite and sulphide of that metal are rarely used in medicine.

The *quantities* used in prescription writing are those of the apothecary, as shown in the typical prescription on p. 1056. The metric system is used exclusively by the physicians of continental Europe, and to a slight extent by American physicians. The quantities are preferably expressed as shown in the prescription given below:

R. Quininæ sulphatis.....	2.00
Ferri reducti.....	1.00
Acidi arsenosi.....	0.05
Misce fiat pilulæ numero xx.	

In this country prescription blanks are sometimes printed with a vertical line intended to separate the grammes from the fractional parts thereof, thus replacing the decimal points in the above prescription, just as in a ledger a line is used to separate dollars from cents.

Rarely does the continental physician write the word "gramme"; quantities, as written above, being clearly understood as meaning the metric system.

As abbreviations of drug names may sometimes lead to dangerous results through misinterpretation, so occasionally the abbreviations for quantities prove a source of error.

It is easy to write the symbol of ounce (℥) when drachm (ʒ) is intended, and there is still another danger lurking in the minim (℥) sign—a misunderstanding that resulted in the death of two persons in New Orleans some fifteen years since.

The minim sign seems distinct enough, but a prominent physician, now dead, gradually modified his minim sign from the correct form to one similar to the symbol for the drachm.



Fig. 232.—Faulty minim sign.

One day the physician wrote the prescription shown above, and the unfortunate druggist, in one of those frightful lapses that might come to any of us, read and dispensed *six drachms* of tincture of aconite instead of the *six minims* desired. Of course

R_x
 Tinct Aconiti ʒvi
 Aquae as ʒi
 Sig. Teaspoonful every 2 hours.

Fig. 233.—Dangerous prescription.

he was to blame, but when we hear of such things we should feel thankful that we were spared the fearful blunder. Naturally, the patient died, and the druggist, completely ruined, after a year of hopeless attempts of retrieving his fortune, gave up the conflict and committed suicide.

The metric quantities as written above are less likely to mislead, although a fly-speck might be mistaken for a decimal point. Another

R For 1849 61 Date 75°
 Ammon. Carbonat ʒi
 Milt. Tuscar ʒi
 Highly Tastespoonful
 3 or 4 times a day for cough

Fig. 234.—Typical prescription.

possibility of danger with physicians unfamiliar with the system is writing ten times the quantity intended. For instance, writing 0.05 (five centigrammes) when 0.005 (five milligrammes) was intended.

Before leaving the subject of the prescription as a document a few typical prescriptions may be of service.

This is a clearly written prescription of a southern practitioner. As already explained, on p. 265, *mistura fusca* is a local synonym for brown mixture.

B

Citrine Ointment 3*ii*
 Ol Bet alb 3*ii*
 Vaseline B 3*ii*
 Msig as directed ex-
 ternally

Fig. 235.—Typical prescription.

This is an equally clear prescription, written by a northern practitioner. What is "Ol. Bet. Alb."?

RL 4820/49, 3301/698
 Lg. Fowler
 Sy Roboran (Pepsin 3viii)
 Sol. Pepsin 3viii
 M. 2 cuillerées à café dans un
 1/2 verre d'eau après chaque repas

Fig. 236.—French prescription.

This, written by a Creole practitioner for a Creole patient, calls for Fowler's solution, a proprietary, "syrupus roborans," and the Solution of Pepsin, U. S. P. 1880. The French directions are translated into English below:

2 cuillerées à café dans un $\frac{1}{2}$ verre d'eau après chaque repas.
 2 teaspoonfuls in half glass of water after each meal.

1.15.91
 Mary Alexander — Alth
 R/ +4909
 254
 Myrte de la mère
 Aride? mende douce
~~are~~ 3H3
 Ate —
 A la friction
 deux fois par jour

Fig. 237.—French prescription.

+49062 354
 Liq. cinchon. cord. (B.) 3jss
 Tr. gelsemini 3j
 Elix. simpl. ad 3ij
 In. & Sig: Une petite
 cuillerée toutes les 3
 heures.

Fig. 238.—Typical prescription.

Figure 237 is written by a Creole physician for an American patient. The French ingredients are: Unguent de la mère (Unguentum Fuscum N. F.) and huile d'amande douce (oil of sweet almond).

Sanitätsrath Dr. Jukes

Hannover

Langelaube 32.

$\frac{1}{2}$ 0,80
 29. Jan. 96
 145.22
 Codein 0,02
 Sacch. alb. 0,5
 Ampul. N. Sol.
 dos. 10, 6
 S. Abstr. 1 p
 f
 An. dring
 0,40
 30% Lösser 250, 0

Fig. 239.—German prescription.

Figure 238 is a clearly written prescription with French directions. Liquor Cinchonæ Cordifoliæ (B) is a special liquid extract of cinchona prepared by Battley and Co., of England. The English translation of directions is given below: Une petite cuillerée toutes les 3 heures. A teaspoonful every 3 hours.

Figure 239 is written by a physician of Germany, calling for six powders (dent. tal. dos.), each containing 2 centigrammes of codeine and a half gramme of sugar. The directions read:

Abends ein Pulver.

Evenings a powder.

It being characteristic of the German prescription that the time of administration is written before the dose.

Dr. Haslinger J. J.

(Belgyógyász és szülész)

Reudel 3-5-ig d. u.

Budapest, IV. k., Kalap-utca 8. I. em. 7.

Rp: Acet. carbol
6.00
Alum. ust.
2.0
Pip. pinit aromat.
2.0
G. dentat.
400.0
Gr. mucron.
40.0
Inf. Gargisme.
5.0
Inf. tint. bellar.
2.0
Inf. muc. ust. cum.
0.02
Alcohol. tinct.
10.0
Inf. 7 drops every 3 hours
1000/15
J. Haslinger

Hungarian No 230.

Fig. 240.—Hungarian prescription.

An interesting illustration of the convenience of the use of Latin in prescriptions is shown when the first and second prescriptions on Fig. 239 are compared. The first, written in Latin, could be compounded by any educated pharmacist in any part of the world. On the other hand, the second, written in German, is apt to prove a puzzler. It reads: 3 per cent. Borwasser, 250 Cc.

That is, a 3 per cent. aqueous solution of boric acid is desired.

The prescriptions on Fig. 240 were written by a German physician residing in Buda-Pest, Hungary, and both can be deciphered by a practiced prescriptionist. For this reason they will not be translated by the writer. Mention, however, might be made of the figures on the right-hand side of the paper. In Germany, Austria, and Hungary, the price of every drug used in prescriptions, as well as the cost of containers and value of time spent in compounding, is carefully regulated by the government, and the figures above mentioned represent the summing up of values from the official schedule.

CHAPTER LVIII

COMPOUNDING OF PRESCRIPTIONS

THE prescription as a document is interesting, but of far greater importance is it that the pharmacist knows how to handle the prescription when it becomes his duty to compound it.

The work of the compounder of the prescription is manifold. He must know how to receive the prescription from and dispense the finished preparation to the customer; he must know how to label the container correctly; he must know how to fill the prescription; he must know how to scan it carefully for errors; and, lastly, he must know how to prepare or put up the medicine called for. To perform each step properly he must be intelligent, alert, tactful, skilful, careful, conscientious, and well informed.

Receiving the Prescription.—As mentioned above, if the prescription does not bear the name of the patient, the compounder should supply the deficiency, explaining it is a part of the system whereby mistakes are prevented. Cases are on record where fatal results have resulted from giving a properly prepared prescription to the person for whom it was not intended. This is most liable to occur if two persons are waiting for prescriptions written by the same doctor—say one for internal use; the other for a liniment. Hence the fact that the pharmacist hands the customer "Dr. Blank's prescription" is not a sufficient safeguard, since there may be two of Dr. Blank's prescriptions being compounded at the same time.

In large stores the giving of checks to the person leaving the prescription is a safeguard against such mistakes. A form for such checks is shown below, the one section being given to the customer, one attached to the prescription, and the third attached to the package (Fig. 241). A simpler method is giving the customer a numbered brass check and writing same number on prescription and package. Safest of all, however, is the securing of name and address of patient.

Dispensing the finished preparation to the customer is brought up chiefly to introduce the subject of the relation of the compounder and dispenser; to emphasize the need of that rare gift, *tact*. This gift cannot be acquired by reading books, and the writer feels he can do no more than suggest the importance of tact on the part of the dispenser by citing a few typical cases.

A woman comes into a store asking for five grains of calomel and five grains of *salt*. The pharmacist, realizing that a prescription had been written and not presented, lest more should be charged for the goods if furnished on a prescription than when sold on verbal order, emphasizes the possible formation of mercuric chloride in such a mixture, tactfully interrogates until the woman admits that a prescription was written, and convinces her that this prescription must have called for calomel and *soda*, and persuades her to bring the prescription.

A man comes in with a printed prescription calling for pills containing several well-known tonics, and also "jerubebbin, 1 grain." Instead of telling the man that the prescription is a fraud (a statement for which the mind of the dupe is prepared by the skilfully worded circular sent by the philanthropist who furnishes prescriptions "free"), the pharmacist quietly brings out Druggists' Circular (1884, p. 138), in which the fraud is exposed. The testimony afforded by this disinterested source convinces even the credulous chap who is saved the dollar he was to send the donor of the prescription "in case the medicine cannot be obtained elsewhere."

A man who once had a prescription wrongly compounded insisted on sitting behind the prescription counter watching the compounder, so as to keep tab. To make things still safer, he usually plied the compounder with questions. The new clerk was a young man just from college; the man, more suspicious than ever, was more persistent in questioning. "What's this medicine?" he inquired, pointing to the then little-known abbreviation "antipy." "Oh," said the youth, nonchalantly, "that's Latin for the new medicine, phenyl-dimethyl-pyrazolon." From that day forth the man considered the youth the smartest and most reliable druggist in town.

GLOBE DRUG CO.
PRESCRIPTION CHECK
261002
Price _____
Dr. _____
Time _____
By No. _____

GLOBE DRUG CO.
PRESCRIPTION CHECK
Price _____
261002

GLOBE DRUG CO.
PRESCRIPTION DEPARTMENT.
WASHINGTON
PLEASE PRESENT THIS CHECK WHEN
CALLING FOR YOUR PRESCRIPTION.
Dr. _____
Time _____
By No. _____
261002

Fig. 241.—Prescription check.

A nervous woman wants to know what a certain prescription she holds in her hand is good for, and what are its constituents. The tactful pharmacist responds that these are questions which the physician who wrote the prescription is alone permitted to answer, and expresses his willingness to place the lady in telephonic connection with the doctor. His kindly offer is declined.

The only other phase of dispensing to be considered is the question of containers and wrapping of the same. This subject is given consideration on pp. 298-300, so here it is only necessary to say that the pharmacist who values his prescription business, picks out the best glassware, finest corks, and most refined boxes and printing.

Labeling the containers is worthy of a special paragraph, as often the effect of the choicest labels is marred by blurred or scratchy, uncouth, or overflourished writing. A severely plain, heavily shaded back hand, written with the blackest ink and allowed to dry without blotting, makes the most attractive form of label, but of late type-written labels have become quite popular. A strikingly written prescription label is given below (Fig. 242). It will be noted that the body of this label contains neither lines nor printing; that the filled-in part of label is a symmetric whole; that the printing is severely plain and as concise as possible. All the data on the label are essential; the number, the name of the physician, the directions, and the date should appear on each label. In some States the law

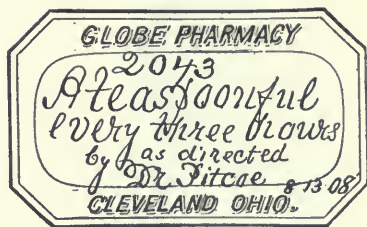


Fig. 242.—Typical label.

directs that the name of the compounder should be put on the container, in which event a small gummed slip, "Dispensed by —," attached to the bottom of the package or bottle usually suffices. It is an excellent practice to have labels intended for external use printed on glazed red paper, this affording a striking contrast to the white paper label for internal preparations. Then, of course, the pharmacist should be provided with slips bearing such frequently used directions "shake well"; "for external use," etc.

Filing Prescriptions.—As the prescription is more or less of a legal document, it is important that the pharmacist carefully preserves either the original or a duplicate. In this country the original is kept by the pharmacist, although the question as to who owns the prescription—the patient or the pharmacist—has been and still is a matter of legal controversy. It would be interesting if the several conflicting court opinions on this matter could be settled for once and all by a decision from the United States Supreme Court. In Germany it is the universal custom to return the prescription to the patient, the pharmacist keeping a carefully checked copy.

The first step toward preserving the prescription for future reference is giving it a number. These numbers run consecutively and are written on the prescription with date and price. One pharmacist known to the writer started new numbering each year, marking the first prescription compounded on January 1st as No. 1, stringing all the prescriptions of one year on a long piece of $\frac{1}{8}$ -inch brass wire, the files of each year being hung on a row of nails driven in the walls, each

mail labeled for the year. This plan is *not* to be commended, for whenever it chanced that the date on the label was faded, it necessitated looking up the same number on possibly a dozen files.

Many large establishments now use consecutive numbering machines, such as the one shown in the appended cut (Fig. 243). Such machines consist of a stamping apparatus, which automatically changes the type of the numbers one unit higher each time an impression is made, thus precluding the possibility of giving two prescriptions the same number. In the more expensive varieties the same number can be repeated in duplicate (or even triplicate), thus enabling the compounder to number the prescription and label before the machine progresses one unit higher. However, the suggestion that the entire label be written renders a duplicate numbering machine unnecessary.

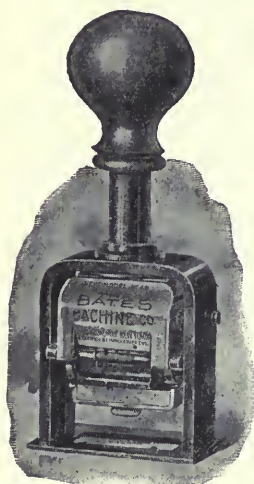


Fig. 243.—Prescription numbering machine.

Several ways of preserving prescriptions are in vogue in this country.

Filing on String or Wire.—This method, outlined just above, is the least satisfactory of all others. The hole in the prescription, when pierced by the wire (or by the needle, if string is used), may cut out a part of the wording most needed. The weight of the pile of prescriptions bearing down and rubbing against a lead-penciled prescription is apt to blur it beyond recognition, while the long file hung on the wall catches dust and soon becomes filthy.

Pasting in a Scrap-book.—This method is quite satisfactory, although the handling and storing of big scrap-books are sometimes inconvenient.

Writing a Copy in a Blank Book and Filing or Returning Originals.—This method, used by all pharmacists of Germany and by a few prescriptionists of America, entails a large amount of work and is apt to lead to errors unless the copy is carefully checked against the original by a second person, as in proof-reading.

Wrapping the Prescriptions in Packages of 100 and Packing Ten Consecutive Hundreds in a Wooden Box.—This method was used by Dr. A. E. Ebert, and there are one or more patented files embodying the same idea on the market. The prescriptions are thus kept perfectly clean, but on opening a package to get the desired prescription, there is danger of the others being mislaid or blown away.

Pasting to a Card which is Filed in a Cabinet.—This method, a result of the modern filing card-cabinet craze, is by all means the most satisfactory method of filing the prescription. The best form of card is that shown below (Fig. 244), with marginal space on which all data can be written, thus leaving the prescription absolutely intact.

In event of success of the present movement looking toward the abolition of the repetition of prescriptions by the pharmacist unless

Necessarily, the druggist must do everything in his power to maintain the most cordial relations with the physician, but this does not mean that he must supinely submit to injustice. A few examples of this phase of the prescriptionist's experience may be of service.

The "blind prescription," the prescription that can be put up only by a few favored druggists, is a nuisance that can and should be immediately stamped out by reporting the offenders—the doctor, to the local medical association; the pharmacist, to the local druggists' association.

Two examples of such blind prescriptions are found on p. 896, in the two names for quinine sulphate. The synonym "sulphatis Americanis Australis" was written in good faith by the physician, who desired to keep the patient from knowing she was taking quinine, and who cheerfully furnished the information to the druggist, while the doctor who perpetrated "sulphat de pelletiere" told the patient the prescription could be prepared only by a druggist blocks away, and furnished the information to the neighboring druggist only after a sharp talk.

A certain "Dr. Jones" wrote a prescription, "Dr. Jones' rheumatic mixture," telling the patient that it could be obtained at a certain remotely situated drug-store. The druggist to whom it was presented called on the doctor and pleasantly asked to be favored with the recipe. It was forthwith furnished with abundant explanations.

Compounding the Prescriptions.—The preparing of each prescription is a law to itself, the blending of the ingredients called for depending entirely on the individual peculiarities of the medicines so blended. In the foregoing pages of this book information has been given of most substances used in American medicine, as well as the methods of compounding various classes of preparations. If this information has been absorbed, the compounding of prescriptions becomes simple—merely the application of principles already learned. At this place, therefore, we need only discuss the technical phases of compounding as it peculiarly applies to the prescriptions, and also the question of incompatibility.

Special Technic of Prescription Compounding.—As just mentioned, the routine compounding of a prescription calling for pills is similar to the directions given for the manufacture of pills on p. 313; for emulsions, as explained on p. 266; and for all other preparations, as outlined in the proper place on the foregoing pages. This done, the compounder should always verify his results by the so-called "*checking system*"; that is, after the prescription is compounded, the finished preparation and the written prescription are handed to the second person, while the compounder repeats the prescription, laying special emphasis on the quantity of each ingredient. This checking system should be used in every pharmacy, no matter how small. If there is but one skilled compounder in the store, he should train the boy to check for him, and if the compounder is by himself, he should check his own work by placing each bottle containing the medicine used in a row on the counter, with the weight employed immediately in front of the bottle. This precaution might be criticized as meaning an outlay for extra weights, but this expenditure is more than compensated for by the security coming from the plan.

In Case of Error.—Perhaps this title seems superfluous, since, of course, no errors should occur, but the writer agrees with an experienced pharmacist who said that whenever he heard of a druggist's mistake he simply thanked God that he was not the offender. Mistakes are liable to occur to all, hence the compounder should guard continually against errors, for even one of these may blast the pharmaceutical reputation of a life-time.

The handling of an error calls for the highest possible tact, hence no fixed and fast rules can be given, except the general statement that an attempt to explain by lying usually makes matters worse. Of course, whenever possible, the error should be repaired without exciting the suspicion of the customer, such as intercepting the messenger or requesting return of medicine, but at all hazards the damage must be repaired even if it is necessary to call the prescribing physician to your aid. This last step, disastrous as it is to the pharmacist, should be taken only in extreme cases.

INCOMPATIBILITIES

The word "incompatibility" means "lack of agreement," and when the several ingredients in a prescription do not agree, such mixture is said to be incompatible. Prescription incompatibility is grouped into three classes, according to the nature of the disagreement; *pharmaceutic* (or physical) incompatibility; *chemical* incompatibility, and *therapeutic* incompatibility. These we will consider in regular order.

Pharmaceutic incompatibility is that form of disagreement depending chiefly on the question of relative solubility. Thus a solution of resinous substance, say, tincture of ginger, will become turbid on addition of water, not because of any chemical change, but because the resin, while soluble in alcohol, is not soluble in alcohol diluted with water. Likewise, a gum in aqueous solution—like mucilage of acacia—will be precipitated on addition of a strongly alcoholic tincture, simply because the gum is insoluble in diluted alcohol. A third typical case of pharmaceutic incompatibility that might be cited is the classic prescription calling for quinine, sulphuric acid, and a licorice preparation (p. 895). The reason for giving this case special emphasis is because, at first glance, it seems a case of chemical incompatibility, since it deals with the active principle of licorice, glycyrrhizin. But since it is based entirely upon the solubility of glycyrrhizin in acid, and not with the chemical decomposition of the glycyrrhizin, it is an example of pharmaceutic incompatibility.

Among pharmaceutic incompatibilities, for the want of a better place, we group such prescription experiences as the dispensing of deliquescent salts, the compounding of pill prescriptions calling for excess of liquid, and those cases where not enough solvent is directed.

Chemical incompatibility is that form of disagreement where a chemical change occurs when two or more substances are blended. Sometimes the chemical change improves the product, and such cases are not considered as incompatibilities. An example of this is the well-known prescription calling for mercuric chloride and potassium iodide (p. 615), when a soluble potassiomeric iodide is formed.

Such cases are rare, however, being limited to the formation of hydroxides from oxides, the neutralization of a base by an acid, and a few special reactions such as the saponification of an oil by addition of alkali.

In the modern conception of solution it is considered that chemical action usually conforms with the following rule:

A chemical reaction occurs between two soluble bodies only when one or more products of such a reaction is either insoluble or gaseous. Always apply the above rule. Thus, if NaCl and KBr be mixed, will there be a reaction? What would be the products of such a reaction? Naught but KCl and NaBr—both soluble bodies—hence no reaction occurs. On the other hand, if we added a solution of NaCl to a solution of AgNO_3 , a reaction would occur because one of the possible reaction products—AgCl—is insoluble.

Hence the whole question hinges on the solubility of the reaction products, and the table of solubilities, given on p. 119, will be of great value in considering this question.

Among the more important cases of precipitation (hence of incompatibility) are the following:

1. *The formation of an insoluble salt, as in the case just cited.*

If Part V., dealing with chemical analysis, has been carefully studied, the most likely forms of such precipitation will have been learned, thus: (a) The alkaline metals, lithium, sodium, potassium, and ammonium, are hardly likely to be precipitated by any chemical (p. 431).

Exception: Potassium salts plus tartaric acid may precipitate potassium bitartrate.

(b) The alkaline earths—barium, strontium, calcium, and magnesium—will be precipitated on addition of carbonates and phosphates.

(c) The third, fourth, fifth, and sixth groups of metals will be precipitated by the carbonates, phosphates, hydroxides, and sulphides.

This statement must be taken in a general way. Thus, with several of the metals, the precipitated hydroxide is redissolved by excess of alkaline hydroxide, and, in truth, arsenic is not precipitated by hydroxide.

(d) The seventh group of metals—silver, lead, and mercuric salts—will be precipitated by all the foregoing, and also by chlorides and sulphates.

Besides these general rules, the special precipitants explained as tests in Part V. will, of course, act as incompatibilities.

2. *Formation of special insoluble organic salts, such as tannate of iron (ink), when tannin-bearing drugs are combined with iron salts; meconate of lead, when opium preparations are combined with lead solutions; and the production of the insoluble blood-red iron salicylate when iron preparations are combined with salicylates.*

3. *Precipitation of alkaloids by alkaloidal reagents* (p. 876). Example of this is the precipitation of the cinchona alkaloids when preparations of this drug are combined with potassium iodide and mercuric chloride.

4. *Formation of insoluble bodies when synthetic organic chemicals are combined with certain reagents.* An example of this is the formation of green crystals when atropine is treated with spirit of nitrous ether that has become acid.

5. *Formation of insoluble bodies when certain organic chemicals are decomposed by certain reagents, such as the decomposition of chloral, by action of alkali, into chloroform* (p. 668).

To give extended examples of these incompatibilities, beyond the outline given above and the mention made of these individual peculiarities in discussion of the chemicals in Parts III. and IV., would take too much space, hence for complete enumeration of incompatibilities the reader is referred to Ruddiman's admirable work on "Incompatibilities," mentioned in Preface.

Incompatibilities due to liberation of gases may be summed up as *explosive prescriptions*, combinations where carbon dioxide is evolved with that mild form of explosion called *effervescence*; and those few cases where gaseous ammonia may be liberated from ammonium salts.

Explosive prescriptions must be known in advance by the compounder, as otherwise an explosion may occur, producing dangerous results. As already explained (p. 450) explosions are produced by a sudden evolution of gases, and all substances liable to produce such gaseous evolution on trituration must be handled with the utmost caution. Already in Parts III. and IV. we have learned most possibilities of this kind, so here a sketchy summary will suffice:

(a) All oxidizing agents, such as potassium chlorate, chromic acid, potassium permanaganate, silver oxide, hydrogen dioxide, and nitric acid, are liable to explode when combined with organic matter, and such oxidizable inorganic matter as sulphur or charcoal.

(b) Hypophosphites are liable to explode when heated above 100°C. , or when combined with any substance (such as *nitrates* and other oxidizing agents) liable to produce phosphine (PH_3).

(c) A modification of "a" is the fact that strong nitric acid and its preparations effervesce with drug preparations containing tannin and with oil of turpentine, sometimes with explosive violence.

(d) Iodine is liable to explode when treated with ammonia or with oil of turpentine.

Effervescent Prescriptions.—When any acid combines with a carbonate, carbon dioxide is evolved with an effervescence which is harmless if allowed to proceed in an open dish. If the ingredients are mixed in a bottle which is immediately corked, the force of the compressed gas is enough to blow out the cork or possibly shatter the bottle. Hence, as already mentioned on p. 444, such mixture should be prepared in graduate or mortar, and the liquid poured into the bottle with only a slight "bead." Aside from a clearly discernible combination of carbonate and acid, look out for prescriptions calling for carbonates and those pharmaceuticals containing acetic acid, such as syrup of squill.

Combinations Liberating Ammonia.—Such prescriptions are rare, being practically confined to combinations of ammonium chloride and lime or of solution of ammonium salts with solution of soda and solution of potassa.

Therapeutic incompatibility is that form of disagreement where the doctor prescribes two medicines that are therapeutically antagonistic. Examples of this are the blending of a stimulant with a sedative, of a mydriatic, like belladonna, with a miotic, like physostigma; of pepsin with alkali, and of pancreatin with acids. The combining of pepsin and pancreatin is also condemned by the best medical authorities.

The question of therapeutic incompatibility is entirely beyond the province of the pharmacist, the writer's experience being that the doctor, no matter how friendly, resents suggestions from the druggist along this line.

Handling Incompatible Prescriptions.—The question of the compounding and dispensing of incompatible prescriptions can best be considered in discussion of each of the several examples of incompatible prescriptions given on the pages which follow.

Discussing the subject in a general way, it can be said that the question—both as to compounding and dispensing—is a matter of good judgment.

Every pharmacist should know an explosive prescription at sight, should know those which should not be combined under any circumstances (such as chromic acid and glycerin), and those which can be mixed if proper precautions are taken (such as mixing nitric acid and oil of turpentine). Explosive prescriptions are a menace to the compounder rather than to the customer, hence self-interest behooves complete knowledge of this class of prescriptions.

In other forms of incompatibility the pharmacist is confronted with two important questions: 1. "Is the prescription safe to dispense?" 2. "How can it be least objectionably compounded?"

As to dispensing, there are frequently cases of *intentional incompatibility*—cases where the doctor really desires an unsightly mixture, because of its therapeutic virtue. We all know that the official chalk mixture is of value because of the insoluble chalk it contains; even so the prescription calling for zinc sulphate and lead acetate must be dispensed containing the precipitate of lead sulphate, since that constitutes a large part of the healing effect of the mixture.

On the other hand, prescriptions in which potent medicines are produced in insoluble form, such as those prescriptions in which powerful alkaloids are precipitated, should be viewed with suspicion by the compounder, and while it is often an exceedingly delicate matter to approach the physician on the subject, it is usually best to let the doctor know, either at the time or later, the possibility of danger. No pharmacist of tact or good sense will bluntly announce to the customer that he will not compound such a prescription, but will always consult the physician. In all cases where this kind of prescription is dispensed, a "shake well" label must be put on the bottle.

Of course, there are prescriptions of this kind which must not be dispensed, such as the mixture of chloral hydrate and an alkali cited above, and in the list of prescriptions which follow mention is made of those which are unsafe to dispense.

A last word on consulting the physician about dangerous incompatibility as well as dangerous doses. Always approach him armed with accepted authority. On dosage, the two dispensatories can be cited, while on incompatibilities, Ruddiman's book is the best guide.

The question as to the best way of compounding an incompatible prescription depends on the individual prescription, and in the line of incompatible prescriptions which follow, the best way of compounding is usually given. In a general way it might be stated that when the two substances likely to react are diluted as much as

possible before mixing, the chances of incompatibility are lessened, as was discussed on p. 390.

Another important question of compounding is, "When is the pharmacist justified in changing a prescription?" The answer is that a change is justified only when such change improves the prescription without affecting its medicinal action. A few examples may be cited:

(a) A prescription calling for a certain alcoholic tincture mixed with gummy substances may precipitate the gum. If the equivalent amount of the fluidextract is used, the precipitation may be avoided.

(b) An aromatic water, when used to dissolve a salt like potassium iodide, has some of its oil separate, and cloudy mixture results. If the salt is dissolved in the smallest quantity of water and then mixed with the aromatic water, no oil will separate.

(d) A prescription called for phenol mixed with an amount of water insufficient to dissolve it. If a part of the water—say one-eighth or one-quarter—be replaced by glycerin, a perfect solution ensues.

(d) Pills calling for ferrous sulphate, U. S. P., or other crystalline salts are found to yield a mass that is too soft. By use of the proportional quantity of exsiccated salt the mass can be easily made. Most of these or similar cases are given among the prescriptions cited on the pages which follow, so this short summary suffices at this place.

Inadvisable is it to make a construction like that cited on p. 1087, that an unsightly prescription calling for "magnesia" can be made sightly by using soluble magnesium sulphate.

In conclusion let it be said that when a change is made, it is always best to let the physician know, explaining why the change was made.

INCOMPATIBLE PRESCRIPTIONS

R. Tinct. iodi.....	℥ij
Liniment. ammoniæ.....	℥iv.
M. Ft. lint.	

Liable to explode, as ammonia combines with iodine, forming the dangerously explosive nitrogen iodide, NI_3 .

R. Spiritus ætheris nitrosi.....	℥ss
Fluidext. uva ursi.....	℥iss.
M. Sig.—Teaspoonful every four hours.	

The spirit of nitrous ether, if acid, is apt to act on the tannin of the uva ursi and froth violently, sometimes exploding the bottle.

R. Acidi chromici.....	gr. x
Glycerini.....	℥ss.

Don't dispense. Dangerously explosive.

R. Olei terebinthinæ.....	℥iv
Acidi nitrici.....	℥iv.

Can be put up if mixed cautiously in large open dish.

R. Argenti oxidi.....	gr. v.
M. Ft. pilulæ No. xx.	

Mass these pills with kaolin and vaseline. Ordinary excipients will cause explosion.

R̄.	Zinci oxidi.....	℥ij
	Zinci carb. præc.....	℥j
	Petrolati.....	℥ss
	Liq. calcis..... ad	℥iv.

This prescription was evidently written with the idea that the fat petrolatum would saponify with the calcium hydroxide as would an animal or vegetable fat. It does not do this, and an unsightly mixture results.

R̄.	Quin. sulphatis.....	gr. x
	Potass. acetatis.....	gr. xx
	Acidi sulphurici.....	gtt. x
	Aquæ..... q.s. ad	℥ij.

Chemical incompatibility. Insoluble quinine acetate formed.

R̄.	Tincturæ cinchonæ.....	℥iss
	Tincturæ guaiac.....	℥ss
	Mucilaginis acaciæ.....	℥iss.

Pharmaceutic incompatibility. Acacia precipitated by the alcohol. Remedy by reducing amount of alcohol by using a proportional quantity of fluidextract of cinchona and enough water to produce bulk equal to amount of tincture called for.

R̄.	Ammonii carbonatis.....	gr. xxiv
	Ferri et ammon. cit.....	℥j
	Glycerini.....	℥ss
	Aquæ..... ad	℥vii.

Chemical. Red ferric hydroxide precipitated by ammonium carbonate.

R̄.	Copaibæ.....	℥j
	Acid. sulph. arom.....	℥ss
	Inf. rosæ..... q.s. ad	℥ij.

Pharmaceutical. Copaiba immiscible. Remedy by emulsifying.

R̄.	Morphinæ sulphatis.....	gr. ij
	Spt. æth. nit.....	℥ss
	Aquæ..... ad	℥vj.

Chemical. Color changes from red to yellow.

R̄.	Hydrarg. chlor. corrosivi.....	gr. j
	Sodii boratis.....	gr. v
	Aqua destillatæ.....	℥iv.

Chemical. When the salts are mixed in concentrated solution, the product is a dirty red color. When in dilute solution it is first colorless, afterward depositing a black powder.

R̄.	Potass. cyanidi.....	gr. j
	Acidi citrici.....	gr. v
	Syrupi pruni virg.....	℥iv.

M. Dessertspoonful.

This is all right, affording a good means of administering HCN, this being liberated in the action of citric acid on the potassium salt.

R̄.	Ammonii chloridi.....	℥j
	Calcis.....	℥iiij
	Aquæ.....	℥ij.

Chemical. $2\text{NH}_4\text{Cl} + \text{Ca}(\text{OH})_2 = \text{CaCl}_2 + 2\text{NH}_3 + 2\text{H}_2\text{O}$. Ammonia gas is generated.

R̄.	Potass. brom.....	℥ij
	Chloralis.....	℥j
	Spts. ammon. arom.....	℥ss
	Syrupi aurant. cort.....	℥ij
	Aquæ..... ad	℥iv.

Chemical. Ammonia in spts. ammon. arom. acts on chloral to form chloroform which is immiscible. Dispense with "shake" label.

R̄. Tincturæ iodi,	}	 āā ʒss.
Collodii,		
Aquæ ammoniæ.		
M. Ft. sol.		

Pharmaceutical. The tincture and the collodion mix to a clear solution, but as soon as the ammonia water is added, the pyroxylin in the collodion is separated in a solid mass. Can be dispensed without altering properties by using spirit of ammonia instead of the water. It is apt to become lighter on standing, due to action of ammonia on iodine. Also a faint possibility of explosion, intimated by comparison with iodine prescription on p. 1081.

R̄. Bismuthi et ammon. cit.....	gr. xxx
Liquor pepsini.....	ʒj
Elix. et aquæ.....	āā ad ʒiv.

Chemical. This solution remains clear only when exactly neutral; the bismuth salt being insoluble in acid and the pepsin insoluble in alkaline solution. It being difficult to preserve neutrality, the bismuth citrate is always precipitated in time.

R̄. Copaibæ.....	ʒiv
Olei cubebæ.....	gtt. xx
Calcis.....	gr. xx.

Chemical. The calx saponifies the copaiba, just as does magnesia in the formerly official mass of copaiba; in this case, however, to make a creamy emulsion.

R̄. Pilulæ colocynthidis comp.....	gr. xx
Hydrargyri chlor. mit.....	gr. vj
Tinct. capsici.....	gtt. xx.

The first ingredient is from the British Pharmacopœia and is essentially our compound extract of colocynth. There is too much liquid and the mass will be too soft. Triturate in warm mortar until the tincture evaporates.

R̄. Vini ipecacuanhæ.....	gtt. xx
Magnesiæ sulphatis.....	gr. xx
Sodii bicarbonatis.....	ʒj
Aquæ.....	ʒj.

Chemical. The two salts effervesce on mixing. Do not bottle until carbon dioxide is all evolved.

R̄. Argenti nitratis.....	gr. v
Micæ panis.....	gr. xx.
M. Ft. pilulæ No. xx.	

Chemical. The salt in the bread may react with the silver nitrate to form insoluble silver chloride.

R̄. Potass. bromidi.....	ʒvj
Aquæ camphoræ.....	ʒvj.
M.	

Pharmaceutical. The soluble salt causes separation of camphor from the water. Accordingly, this must be dispensed with a "shake" label.

R̄. Tincturæ nucis vomicæ.....	ʒss
Tincturæ cinchonæ comp.....	ʒiiss.

Pharmaceutical. Fat in tincture of nux vomica precipitated by the hydro-alcoholic tincture of cinchona, causing milkiness.

If, however, the tincture of nux vomica be that of U. S. P. VIII., prepared from the extract, the difficulty will be obviated.

R̄.	Syrupi ferri iodidi.....	℥iss
	Potassii iodidi.....	℥j
	Potassii carbonatis.....	℥iss
	Syrupi tolutani.....	℥ss
	Aquæ.....	℥ij.

Chemical. The carbonate reacts with ferrous iodide, forming insoluble ferrous carbonate, $K_2CO_3 + FeI_2 = 2KI + FeCO_3$.

R̄.	Quininae bisulphatis.....	℥ss
	Misturæ Bashami.....	℥ij.

Chemical. The free acetic acid in the Basham's mixture will precipitate insoluble quinine acetate.

R̄.	Potassii bromidi.....	℥iv
	Aquæ menthæ piperitæ.....	℥vj.

Pharmaceutical. The bromide, in dissolving, refrigerates the peppermint water, rendering the oil dissolved less soluble and causing excess to separate, making milky mixture. Remedied by dissolving the salt in pure water and adding peppermint water to the required amount.

R̄.	Potassii bicarbonatis.....	℥j
	Tinctura guaiaci ammoniatæ.....	℥ij
	Mucilaginis acaciæ.....	℥ij
	Quininae sulphatis.....	gr. viij
	Acidi sulphuric. arom.....	℥iv
	Aquæ.....	ad ℥iv.

Chemical. The excess of bicarbonate will precipitate the quinine, the resin in the tincture of guaiac will be thrown out by the water. No remedy.

R̄.	Tincturæ guaiaci ammoniatæ.....	℥ij
	Spiritus ætheris nitrosi.....	℥ss
	Aquæ cinnamomi.....	ad ℥iv.

M.

Pharmaceutical. Resin of guaiac precipitated by the water. Remedy by emulsionizing.

R̄.	Asafetidæ.....	gr. xv
	Pulveris camphoræ.....	gr. x
	Terebinthinæ albæ.....	gr. vj
	Olei menth. pip.....	gtt. iv.

M. Ft. pil. No. x.

By warming slightly, a plastic mass may be obtained.

R̄.	Cinchoninae sulphatis.....	℥iij
	Acidi nitrici.....	gtt. x
	Aquæ.....	℥iv.

M.

Pharmaceutical. Amount of acid not sufficient to dissolve the cinchonine, hence more is to be added.

R̄.	Croton chloralis.....	℥iv
	Alcoholis, q.s. ft. sol.	
	Mucilaginis acaciæ.....	℥ij.

Pharmaceutical. The amount of alcohol necessary to dissolve the croton chlora will be enough to precipitate the acacia from the mucilage, so leave out the alcohol.

R̄.	Potassii cyanidi.....	gr. j
	Spiritus chloroformi.....	℥iss
	Fluidext. pruni. virg.....	℥ij
	Tincturæ opii camph.....	℥ss.

This is miscible.

R.	Tincturæ krameriae.....	℥ij
	Tincturæ opii.....	℥iv
	Aquæ camphoræ.....	℥iss
	Aquæ menthæ piperitæ.....	℥j.

Chemical. The tannin in the tincture of krameria will precipitate the morphine.

R.	Calc. hypophosphitis.....	℥iv
	Liquor potassii arsenitis.....	℥j
	Tinctura ferri chloridi.....	℥j
	Strychninæ sulphatis.....	gr. j
	Acidi phosphorici dil.....	℥j
	Aquæ.....	ad ℥iv.

Chemical. The arsenous acid of Fowler's solution produces, with tincture of ferric chloride, insoluble ferric arsenite.

R.	Ferri et quininæ citratis.....	℥iv
	Tincturæ valerianæ ammoniatæ.....	℥vj
	Aquæ.....	q.s. ℥xij.

Chemical. The ammonia will precipitate the quinine from the scale salt solution.

R.	Potassii bicarbonatis.....	℥j
	Potassii salicylatis.....	℥j
	Spiritus chloroformi.....	℥j
	Aquæ.....	q.s. ad ℥viiij.

Pharmaceutical. Spirit of chloroform is immiscible with that quantity of water, and a milky mixture results on agitation, the chloroform separating on standing.

R.	Tincturæ opii.....	℥ss
	Liq. plumb. subacet. dil.....	q.s. ad Oj.

Chemical. A popular lotion that must be dispensed with a "shake" label. Lead meconate precipitated.

R.	Hydrargyri chloridi corros.....	gr. ij
	Liq. calcis.....	℥xij.

Chemical. The popular and former official, "yellow wash," lime water reacting with mercuric chloride to form insoluble yellow mercuric oxide by the following reaction: $\text{HgCl}_2 + \text{Ca}(\text{OH})_2 = \text{HgO} + \text{CaCl}_2 + \text{H}_2\text{O}$. Dispense with "shake" label.

R.	Quininæ sulphatis.....	gr. xxx
	Tincturæ ferri chloridi.....	℥j
	Syrupi et aquæ.....	ad ℥j.

Chemical. The quinine will not entirely dissolve, there being an insufficient amount of acid in the tincture to accomplish the purpose. Add a few drops of hydrochloric acid.

R.	Quininæ hydrochloratis.....	3.0
	Antipyrine.....	2.0
	Aquæ destillatæ.....	5.0

Pharmaceutical. The usually insoluble quinine salt will, under the influence of antipyrine, dissolve in the small quantity of water directed in the above prescription, which is lately introduced as "chinopyrine," as a good method of administering quinine hypodermically.

R.	Hydrarg. chlor. mitis.....	gr. xx
	Antipyrine.....	gr. xl.
	M. Ft. pulv. No. xx.	

Chemical. Dangerous, as corrosive sublimate is formed by the following reaction: $3\text{Hg}_2\text{Cl}_2 + \text{H}_2\text{O} + 2\text{C}_{11}\text{H}_{12}\text{N}_2\text{O} = 2\text{Hg} + \text{Hg}_2\text{O} + 2\text{HgCl}_2 + 2\text{C}_{11}\text{H}_{12}\text{N}_2\text{OHCl}$.

- ℞. Fluidext. hamamelidis..... ʒij
 Fluidext. hydrastis..... ʒiv.
 M.

Chemical. Precipitates when combined. Probably due to the action of tannin in the hamamelis on the hydrastis alkaloids.

- ℞. Hydrargyri chloridi corrosivi..... gr. ʒ
 Strychninæ sulphatis..... gr. j
 Quininæ sulphatis..... ʒj
 Liquor acidi arsenosi..... ʒj
 Tincturæ ferri chloridi..... ʒij
 Misturæ ferri et ammon. acet..... ad ʒiv.

Chemical. Clear solution is formed by adding the tincture of iron and the solution of arsenous acid to about three ounces of freshly prepared Basham's mixture, dissolving the alkaloids in this mixture, adding an aqueous solution of the corrosive sublimate, and finishing with sufficient Basham's mixture to make four fluidounces. But the alkaloids are precipitated by the corrosive sublimate on standing, and the mixture is too dangerous to dispense.

- ℞. Tincturæ myrrhæ..... ʒij
 Morphinæ acetatis..... gr. j
 Acidi tannici..... ʒss
 Syrupi Zingiberis..... ʒj.
 M. Ft. sol.

Both *Chemical* and *Pharmaceutical*. Tannin precipitates the morphine. Resin of myrrh separated by the syrup. Nothing can be done with it.

- ℞. Hydrargyri bichloridi..... gr. j
 Liq. potass. arsenitis..... gtt. xlvij
 Syrupi ferri iodidi..... ʒvj
 Elixir cinchonæ..... ad ʒvij.

Chemical. Almost anything is possible. Thus the bichloride separates mercury when combined with the potassium bicarbonate of Fowler's solution; it is incompatible with ferrous iodide and precipitates the alkaloids of the cinchona elixir, while the syrup is incompatible with everything else. As the "Pharmaceutical Era" (from which it is taken) says: "A much more incongruous mixture can hardly be devised."

- ℞. Cocainæ hydrochlor..... gr. ij
 Sodii boratis..... gr. x
 Aquæ..... ʒj.
 M. Ft. sol.
 Sig.—Apply to eye.

Cocaine or any other alkaloid will be precipitated by the alkaline borax. If boric acid ~~was~~ used, no precipitation would occur.

- ℞. Hydrarg. bichlorid..... gr. j
 Potassii iodidi..... ʒij
 Tincturæ cinchonæ comp..... ʒvij.
 M. Ft. sol.
 Sig.—Teaspoonful thrice daily.

The bichloride and the iodide combine to form potassio-mercuric iodide, an alkaloidal precipitant (p. 876). This will throw the cinchona alkaloid out of solution.

- ℞. Ammonii muriatis..... ʒj
 Potassii iodidi..... ʒss
 Tincturæ opii camph..... ʒss
 Syrupi pruni virg..... ʒij
 Syrupi tolutani..... ʒij
 Aquæ..... q.s. ad ʒvj.

Chemical. The iodide will precipitate some of the constituents of the paregoric and the syrup.

R̄.	Fluidext. nucis vomicæ.....	m̄xl
	Fluidext. digitalis.....	℥iiss
	Tincturæ strophanthi.....	℥ss
	Potassii nitratis.....	℥iiss
	Elixir gentianæ et ferri.....	ad ℥vj.

Chemical. After standing twenty-four hours bottle containing this mixture exploded. Explosion due to decomposition of the nitrate by tannin and resinous matters in the fluidextracts; large quantities of nitrogen oxides being liberated. Do not dispense.

R̄.	Argenti nitratis.....	gr. ½
	Bismuthi subnitratis.....	gr. ij
	Ext. belladonnæ fol.....	gr. ʒss.
	Dent. tales dos. No. x.	

Chemical. Incompatible, though often prescribed with good results. Silver salt reduced by vegetable extracts. Dispense by adding a grain of kaolin to each pill and making up with petrolatum. This will lessen likelihood of reduction, although not totally removing possibility.

R̄.	Quinina bisulphatis.....	℥j
	Sodii hyposulphitis.....	℥iiss
	Glycerini.....	℥j
	Aquæ cinnamomi.....	ad ℥iv.

M.

Sig.—Shake and take a teaspoonful one hour before meals, in water.

Chemical. The hyposulphite reacts with the precipitation of sulphur and the evolution of sulphurous acid gas. The sulphur precipitates in varying tints, from yellow, through pink, to gray, causing much annoyance in arousing suspicions of the patient. It is to be dispensed, however, as it possesses therapeutic value.

R̄.	Potassii iodidi	}.....	āā ℥ss.
	Liquor potassii arsenitis		

Chemical. Precipitate of oxyiodides of arsenic formed.

R̄.	Potassii citratis.....	℥iij
	Potassii bicarbonatis.....	℥iij
	Tincturæ belladonnæ.....	℥iiss
	Aquæ menthæ piperitæ.....	q.s. ad ℥viij.

Chemical. The salts effervesce on combining, and a precipitation occurs when the tincture is added. The effervescence is probably due to the following reactions: $K_3C_6H_5O_7 + KHCO_3 + H_2O = K_2HC_6H_5O_7 + CO_2 + 2KOH$. And the alkaline hydroxide precipitates the alkaloids from the tincture. A dangerous mixture that must be dispensed with a "shake" label.

The following prescriptions were submitted by W. F. Kaemmerer, at the meeting of the American Pharmaceutical Association held in 1905, and were commented upon by a dozen prominent pharmacists in the "American Druggist" (November 27, 1905). The criticisms given below represent the consensus of opinion of these authorities.

R̄.	Ferrous sulphate.....	5 grains
	Magnesia.....	10 grains
	Peppermint water.....	11 fluidrachms
	Spirit of nutmeg.....	1 fluidrachm.

Mix. Of such dose, make 1 pint.

Directions.—One teaspoonful twice a day.

Would produce an unsightly mixture, since magnesia is insoluble. Mr. Kaemmerer substituted magnesium sulphate, thereby obtaining a clear liquid, a step approved by three gentlemen consulted and condemned by six.

- R̄. Bromine ointment, ten per cent. 1 ounce.
Use as directed.

Mr. Kaemmerer put it up like an ointment of iodine, using potassium bromide to make the bromine soluble. Proportions: bromine, 50 grains; potassium bromide, 100 grains; water, 150 grains; anhydrous wool-fat, 200 grains. Avoid disagreeable fumes of bromine by surrounding mortar with dishes containing ammonia water, suggests Mr. Dunning.

- R̄. Calcium sulphide..... 15 grains.
Make into 60 pills.

Kaemmerer made as quarter-grain granules, using glucose as excipient. Five of the gentlemen consulted thought enough sugar of milk or other diluent should be added to make a one-grain pill, and three advised coating with gelatin or balsam of tolu.

- R̄. Calomel..... 1 grain
Urotropin..... 1 drachm.
Mix and divide into 12 powders.
Directions.—One every four hours.

Mr. Kaemmerer declares that the combination is incompatible, but Messrs. Hynson and Dunning claim that if the urotropin is dry and pure, the reaction between the two is insignificant.

- R̄. Potassium bicarbonate..... 3 drachms
Potassium citrate..... 1 ounce
Tincture hyoseyamus..... 3 fluidrachms
Syrup of Tolu..... q.s. to make 3 fluidounces.
Directions.—Teaspoonful every two hours.

Effervescence occurs, due not to acidity of the citrate, but to the acids (benzoic and cinnamic) of the syrup of Tolu.

- R̄. Sodium phosphate..... 2½ ounces
Sodium bromide..... ½ ounce.
Directions.—Teaspoonful four times a day in water.

Triturates to pasty mass, due to the 60 per cent. water in the official crystalline sodium phosphate. Best remedy is that suggested by Mr. Hankey, of using exsiccated sodium phosphate, 1½ ounces, and sugar of milk, 1½ ounces.

- R̄. Gold bromide..... 6 grains
Water..... 6 fluidrachms.
Make a solution.

Mr. Kaemmerer states the salt does not dissolve to a perfectly clear solution. He therefore added a small amount of potassium bromide, a step approved by all consulted, although Mr. Hankey emphasizes that chemically pure gold bromide dissolves to clear solution.

- R̄. Mercuric chloride..... q.s.
Petrolatum..... 2 drachms.
One in ten thousand.

Merely a problem in arithmetic, and the application of common sense in dispensing:

2 drachms of a 1:10,000 ointment means $120 \times \frac{1}{10000} = \frac{120}{10000} = \frac{12}{1000} = \frac{3}{250}$, or about $\frac{1}{80}$ grain of bichloride. To weigh this small amount is practically impossible on the average prescription scale, and to secure the amount it is necessary to follow some procedure like that of Mr. Kaemmerer, who made an aqueous solution containing 1 grain of bichloride to 1000 minims, and mixing 12 minims of this solution with the petrolatum. Mr. Beringer objects to the use of water, and instead makes an ointment containing 1 grain of bichloride and 99 grains of lanolin and then mixes 1.2 grains of this with petrolatum.

R̄.	Quinine sulphate.....	3 drachms
	Diluted sulphuric acid q. s. to dissolve	
	Sodium salicylate.....	4 drachms
	Extract of malt.....q.s. ad	12 fluidounces.
	Directions.—Teaspoonful three times a day.	

As in the quinine, sulphuric acid, glycyrrhiza mixture, the acid is best omitted. This is agreed to by all consulted.

R̄.	Ammonium iodide.....	1 drachm
	Apomorphine.....	1 grain
	Diluted hydrochloric acid.....	10 minims
	Syrup of licorice.....	1 fluidounce
	Peppermint water.....	2 fluidounces.
	Directions.—Teaspoonful every three or four hours.	

Mr. Kaemmerer omitted the acid to prevent separation of glycyrrhizin. Mr. Dunning calls attention to the possible danger of dispensing the powerful emetic apomorphine in insoluble form, and advises the use of the hydrochloride, thus doing away with the need of the acid.

R̄.	Salicylic acid.....	3 drachms
	Potassium acetate.....	4 drachms
	Glycerin.....	1 fluidounce
	Water.....	4 fluidounces.
	Directions.—Teaspoonful four times a day.	

This makes a clear solution, alkaline acetates having the property of dissolving considerable salicylic acid.

R̄.	Salicylic acid.....	2 drachms
	Solution of ammonium acetate.....	2 fluidounces
	Spirit of nitrous ether.....	$\frac{1}{2}$ fluidounce.
	Syrup.....	$\frac{1}{2}$ fluidounce.
	Directions.—Two teaspoonfuls every three hours until relieved.	

The acid will be largely dissolved by the alkaline solution of ammonium acetate. There is a possibility that the salicylic acid may react on the spirit of nitrous ether, especially if it is acid. Kaemmerer filtered out excess of salicylic acid, a procedure not approved by most of the gentlemen consulted; Mr. Hankey suggesting that enough ammonia be added to insure complete solution.

R̄.	Potassium citrate.....	1 ounce
	Ammonium benzoate.....	4 drachms
	Water.....q.s. ad	4 fluidounces.
	Directions.—Teaspoonful every three hours.	

Although Mr. Kaemmerer reported that the combination did not make a clear solution, the majority of those consulted agreed that if the official salts are used, a clear solution will result. Mr. Dunning calls attention to the fact that the acid ammonium benzoate is only sparingly soluble, and that the official salt on standing loses ammonia, forming the acid benzoate. Therefore, if a cloudy mixture results, just enough ammonia should be added to insure solution.

R̄.	Morphine sulphate.....	3 grains
	Potassium cyanide.....	4 grains
	Aromatic sulphuric acid.....	2½ drachms.
	Syrup of wild cherry.....q.s. ad	4 fluidounces.
	Directions.—Teaspoonful three times a day.	

This prescription is entirely safe, even though hydrocyanic acid is liberated by the sulphuric acid. It is merely fortifying the hydrocyanic acid content of the syrup.

- ℞. Magnesium sulphate..... 3 ounces
 Aromatic sulphuric acid..... 2½ fluidounces.
 Syrup.....q.s. ad 8 fluidounces.
 Directions.—Teaspoonful two or three times daily in water.

The problem is whether three ounces of magnesium sulphate will dissolve in the amount of syrup. Mr. Kaemmerer dissolved it in three ounces of water, and then added syrup to make eight ounces. Others advised dissolving the salt in the syrup by use of gentle heat.

- ℞. Fluidextract of digitalis..... 1 fluidrachm.
 Put into 20 capsules.
 Directions.—One every three hours.

Mr. Kaemmerer mixed the fluidextract with magnesium carbonate and massed with glycerite of tragacanth. Several objected to the use of magnesium carbonate lest its alkalinity decompose the delicate constituents of digitalis, advising the use of sugar of milk as a diluent.

- ℞. Creosotal..... 1½ fluidrachms.
 Basham's mixture..... 4 fluidounces.
 Directions.—Two teaspoonfuls after each meal.

Creosotal will not dissolve in aqueous liquids, so Mr. Kaemmerer emulsified with 1 drachm of acacia and 2 drachms of water and then added Basham's mixture. Mr. Hynson gives the good suggestion to prepare two ounces of a double strength Basham's mixture, thus making it possible to prepare a two-ounce emulsion of the creosotal.

- ℞. Tincture of chloride of iron..... 1 fluidounce
 Diluted phosphoric acid..... 2 fluidounces
 Glycerin..... 2 fluidounces
 100 per cent. solution of sodium phosphate q.s. 8 fluidounces.
 Directions.—Teaspoonful in a half-glass of water after each meal.

If glycerin, solution of sodium phosphate, and a mixture of the acid and tincture of ferric chloride are mixed, a creamy mixture results. If glycerin, the acid, the tincture, and then the sodium phosphate solution are mixed and then warmed, a clear, light greenish solution results. In order to make a clear liquid, the solution of sodium phosphate (which is now official as *Liquor Sodii Phosphatis Compositus*) must contain the pharmacopœial amount of citric acid.

- ℞. Iron pyrophosphate..... 2 drachms
 Diluted phosphoric acid..... 3 fluidrachms
 Syrup.....q.s. ad 3 fluidounces.
 Directions.—Teaspoonful in water an hour after meals.

If carelessly compounded, the ferric pyrophosphate will be precipitated by the acid. Mr. Hankey says if the pyrophosphate will be dissolved in 3 fluidrachms of hot water, the syrup then added, and lastly the acid, little by little, shaking after each addition, a clear solution results. Mr. Kaemmerer used Diluted Metaphosphoric Acid N. F. (p. 429) instead of the official diluted phosphoric acid.

- ℞. Hyoscine hydrobromate..... 2 grains.
 Make 21 pills.
 Directions.—One after each meal.

As this stands, the prescription calls for an excessive dose of the alkaloid. As to compounding, use enough of a diluent (Mr. Kaemmerer used arrowroot) to make a pill weighing about 1 grain.

PART VII

LABORATORY EXERCISES

INTRODUCTION

ESSENTIAL to the making of an allround pharmacist is the ability to carry out pharmaceutic operations, and any thorough course of pharmaceutic instruction must include laboratory exercises as well as theories. The following line of laboratory work has been used with success by the writer for the past decade, and in order to secure best results, every step directed on the following pages should be followed by the student, *particularly the calculations, which are intentionally left incomplete*. To carry out these exercises, the student should have access to a laboratory, that is, a room provided with a working table, provided with gas, Bunsen burners (p. 71), water-bath, (p. 75), filtering or retortist and, with appropriate movable rings (p. 98), and a Liebig condenser (p. 97). Every drug-store should possess such a working table and appliances.

The special apparatus needed to carry out this work is as follows, and should be a part of the equipment of every well-conducted pharmacy:

- | | |
|--|--|
| 1 thermometer: Centigrade markings on one side; Fahrenheit on the other, markings to 250° C. | 1 8-inch spatula. |
| 1 pyknometer of about 25 Cc. capacity. | 1 4-oz. funnel. |
| 1 set of hydrometers; one, heavy Baumé degrees; the other, light Baumé degrees. | 1 2-oz. funnel. |
| 1 hydrometer cylinder. | 1 4-oz. beaker. |
| 1 alcoholometer. | 1 2-oz. beaker. |
| 1 set Lovi's beads. | 2 1-pint Florence flasks. |
| 1 Mohr-Westphal balance. | 1 4-oz. Erlenmeyer flask. |
| 1 prescription balance. | 1 piece wire gauze for heating, 6 by 6 inches. |
| 1 less accurate scale. | 1 16-oz. porcelain evaporating dish. |
| 1 package (100) 4-inch <i>white</i> filter-paper. | 2 2-oz. evaporating dishes. |
| 2 packages (200) 6-inch <i>white</i> filter-paper. | 1 glass rod, 3 feet long (to be cut into lengths). |
| 1 small rolling-pin. | 1 length, about 3 feet, glass tubing. |
| 1 lozenge cutter. | 1 8-oz. Wedgewood mortar. |
| 1 suppository mold (for 6 suppositories). | 1 1-pint glass percolator. |
| 1 suppository compressing machine. | 2 8-oz. wide-mouthed bottles. |
| 1 tin plaster dipper. | 1 pill tile, 6 by 6 inches. |
| | 1 60 Cc. graduate. |
| | 1 set of weights, 50 Gm. down. |
| | 24 test-tubes, 6 by $\frac{1}{2}$ inches. |

Of this list, the Baumé hydrometers, Lovi's beads, Mohr-Westphal balance, and suppository compressing machine, since expensive and not absolutely essential, can be omitted. The set from 8-inch spatula on can be purchased for about seven dollars.

The material needed for the experiments is:

2 oz. copper sulphate.	2 oz. oil of sweet almond.
2 oz. alum.	1 oz. rose water.
$\frac{1}{2}$ lb. ferrous sulphate.	1 oz. powdered borax.
$\frac{1}{4}$ lb. sodium sulphate.	$\frac{1}{2}$ oz. mercury.
1 oz. card teeth.	1 oz. rose honey.
1 lb. sugar, granulated.	4 oz. cod liver oil.
1 oz. benzoin.	1 oz. rectified oil of turpentine.
1 oz. camphor.	1 oz. unslaked lime.
1 oz. zinc oxide C. P.	4 oz. ammonia water, 10 per cent.
1 lb. gentian, No. 30 powder.	1 lb. sulphuric acid C. P.
$\frac{1}{8}$ oz. acetanilide.	1 oz. potassium iodide.
$\frac{1}{8}$ oz. compound extract of colocynth.	1 oz. chlorinated lime.
$\frac{1}{8}$ oz. calomel.	$\frac{1}{2}$ oz. magnesium carbonate.
$\frac{1}{8}$ oz. resin of jalap.	2 oz. citric acid.
$\frac{1}{8}$ oz. powdered gamboge.	1 oz. sodium bicarbonate.
$\frac{1}{8}$ oz. phosphorus.	$\frac{1}{2}$ oz. mossy zinc.
1 oz. powdered althæa.	$\frac{1}{4}$ lb. hydrochloric acid.
1 oz. powdered glycyrrhiza.	$\frac{1}{4}$ lb. nitric acid.
1 oz. powdered acacia.	$\frac{1}{8}$ oz. precipitated zinc carbonate.
$\frac{1}{4}$ lb. chloroform.	1 oz. honey.
2 oz. powdered potassium chlorate.	$\frac{1}{4}$ oz. iodine.
1 oz. confectioner's sugar.	2 oz. lead acetate.
1 oz. powdered tragacanth.	1 oz. litharge.
$\frac{1}{8}$ oz. soft extract of belladonna.	2 oz. olive oil.
2 oz. butter of cacao.	1 oz. rosin.
$\frac{1}{2}$ oz. castor oil.	1 oz. yellow wax.
$\frac{1}{4}$ lb. glycerin.	$\frac{1}{2}$ oz. corrosive sublimate.
2 oz. monohydrated sodium carbonate.	$\frac{1}{4}$ lb. lard.
$\frac{1}{8}$ oz. stearic acid.	$\frac{1}{2}$ oz. bismuth subnitrate.
1 oz. spermaceti.	$\frac{1}{2}$ oz. white castile soap.
1 oz. white wax.	

CHAPTER LIX

GALENIC PREPARATIONS

THERMOMETER TESTING

Boiling-point of Water.—Take two thermometers, one with Fahrenheit and the other with Centigrade scale. Suspend each in pint flask, which should rest on wire gauze or ring, high enough to permit a flame of Bunsen burner underneath. Have in the flask about 150 Cc. water, and do not let the thermometers dip below the surface of the water.

Apply heat, and when water boils and a goodly amount of steam is passing from the flask, read the height of mercury in each thermometer. The Fahrenheit will read about 210°; the Centigrade, about 98°. If much below these points, it is likely due to part of the thermometer being outside the flask and beyond influence of steam zone. To insure full action of steam, the neck of the flask may be lengthened by means of a roll of stiff paper or by inverting over flask a narrow glass percolator. The latter method is, however, not highly satisfactory, first because of danger of cracking percolator; second, because the condensed steam drops on outside of flask.

Note.—Water boils at 212° F. or 100° C. at *sea-level*, or at the atmospheric pressure equal to 760 millimeters of mercury. (See p. 86.) If the thermometer is tested in a high building or at any place higher than sea-level, the boiling-point will be less than the standard.

There is no mathematic formula expressing relation of boiling-point to atmospheric pressure which can be considered a true law, but Regnault worked out a table of boiling-points under different pressures which can here be condensed to the following four lines:

<i>Barometric pressure.</i>	<i>Boiling-point of water.</i>
91.92 mm.	50° C.
525.45 mm.	90° C.
760.00 mm.	100° C.
1520.00 mm.	120.4° C.

To this can be added the statement that an ascent of 1080 feet makes a decrease in the boiling-point of water of 1° C.

Melting-point of Ice.—Put four-ounce funnel into iron ring attached to filtering stand and place two-ounce beaker beneath it. Break ice into pieces about size of a pea, and fill funnel with same. Insert in midst of ice the two thermometers used in boiling-point experiment, and let stand five or ten minutes; then take out and read quickly. The Fahrenheit thermometer should read 32 degrees—the Centigrade, zero degrees.

SPECIFIC GRAVITY OF LIQUIDS

First, estimate specific gravity as follows:

Take a one-ounce graduate and carefully tare it. “Tare” means to counterbalance it—to put on the other scale just enough weights so that it will be exactly balanced.

Now, very carefully measure into it exactly one fluidounce of water (distilled, if possible) and note how many grains the water weighs.

Then weigh in similar manner one fluidounce of alcohol. Estimate its specific gravity by comparison of its weight with the weight of the fluidounce of water, and then looking in the back of the pharmacopœia or dispensatory, find the table showing alcoholic strength according to specific gravity (commonly called alcohol table), and deduce from the specific gravity what the strength of the alcohol is.

Lastly, find the weight of a fluidounce of glycerin and work out its specific gravity from this weight, remembering always that specific gravity is the relative weight of a body as compared to the weight of an equal bulk of water.

Then do same work more accurately by means of pycnometer. (See p. 48.) The necessary data to be obtained are as follows:

<i>Water.</i> —Weight of pycnometer	_____
Weight of pycnometer filled with water	_____
A. Weight of <i>water</i> held by pycnometer	_____
<i>Alcohol.</i> —Weight of pycnometer	_____
Weight of pycnometer filled with alcohol	_____
B. Weight of <i>alcohol</i> held by pycnometer	_____
<i>Glycerin.</i> —Weight of pycnometer	_____
Weight of pycnometer filled with glycerin	_____
c. Weight of <i>glycerin</i> held by pycnometer	_____

Calculations.—Since the same pyknometer is used in each case, and since specific gravity means how many times heavier than water is a certain substance, we find the specific gravity of alcohol by dividing weight of alcohol by weight of same volume water:

$$\frac{B}{A} = \text{sp. gr. of alcohol;}$$

and we find the specific gravity of glycerin by dividing its weight by weight of same volume of water.

$$\frac{C}{A} = \text{sp. gr. of glycerin.}$$

Oil of Turpentine.—Lastly, fill pyknometer with oil of turpentine, and from the weight of turpentine deduce the specific gravity of that liquid, viz.:

Weight of pyknometer filled with turpentine	÷	weight of pyknometer filled with water	=	specific gravity.
				(How many times the oil of turpentine is heavier than water.)

Be sure to wash the pyknometer carefully after the turpentine experiment until free from the odor of that oil.

Remarks.—In weighing, give value to centigrammes.

Use *distilled* water in all specific gravity experiments. In weighing the empty pyknometer, be sure it is absolutely dry (wipe it with clean handkerchief) both inside and out.

In filling it with any liquid be sure to have no air-bubbles in the liquid and to have it completely filled up to top of capillary orifice in stopper. The latter is accomplished by filling almost to top of neck of pyknometer, and then suddenly and evenly plunging the stopper into the neck. The excess of liquid passes through capillary to top of stopper, and is carefully wiped off with a piece of filter-paper. The entire pyknometer is then carefully wiped with a clean handkerchief and then weighed.

In wiping the pyknometer after wiping tops of stopper the heat of hand may cause expansion of liquid, which will rise through capillary to top of stopper. Do not wipe off this excess, but weigh it on top of stopper.

After weighing, empty out liquid and carefully wash out pyknometer with water (and alcohol if necessary), and dry perfectly before filling with another liquid. Water, if present, will contaminate the other liquid.

In accurate work the temperature at which pyknometer is filled should be noted, and, if possible, the liquid should be at 25° C.

SPECIFIC GRAVITY OF LIQUIDS WITH HYDROMETER

Take three tall cylinders and fill about three-quarters—the first with water, the second with alcohol, the third with glycerin.

Take two hydrometers (p. 49), one for heavy liquids, the other for light liquids, and note to which degree the hydrometer sinks in each liquid—the line just appearing at the surface of liquid.

	Degrees light.	Degrees heavy.
Water	-----	-----
Alcohol	-----	-----
Glycerin	-----	-----

These are "degrees Baumé," and calculate therefrom by process given on p. 50 the specific gravity of each liquid. From the specific gravity of the alcohol deduce by means of alcohol table given in the pharmacopœia its alcoholic percentage.

Remarks.—Be sure that each cylinder is perfectly clean and dry before pouring liquids in, so that each liquid can be returned to container after the estimations, absolutely pure. In the same way carefully clean and dry each hydrometer before transferring to another liquid.

Avoid air-bubbles in putting hydrometers into liquids, as air is lighter than the liquids and air-bubbles vitiate results.

As the principle of the action of the hydrometer is fully explained on p. 49, nothing but the actual operation is here given.

With Alcoholometer.—Take very tall cylinder,—a long narrow percolator with cork in neck does quite nicely at a pinch,—and fill about nine-tenths with alcohol. Sink therein the alcoholometer (p. 50) and note point at which it floats. Note the two scales on the alcoholometer—one the alcoholic percentage, the other, its value in "proof." For "proof" valuation see p. 657.

With Lovi's Beads.—Throw a handful of Lovi's beads into water in a pint graduate. Some will sink, some will float on surface, and perhaps one will float midway in liquid. Take out those on the surface and all will be found to have specific gravity less than water; those which have sunk have specific gravity greater than water, while the one floating will be the specific gravity of water. Carefully dry and repeat with any other liquid if desired.

With Mohr's Balance.—Take Mohr's balance (p. 52) and note that the sinker in air is exactly counterpoised by weight on other end. Put water into cylinder, and immerse sinker therein. Note that the buoyant force of the water presses sinker upward. Place largest weight on notch directly over place from which sinker is suspended, when equilibrium will be restored. If desired, the *volume* of the sinker can be measured by sinking it into water contained in a burette and noting rise of water; the *weight* of weight which restores equilibrium may then be taken, in which event it will be found that the *volume* of the sinker, expressed in cubic centimeters, is just the same as number of grammes that the weight weighs.

(The portion of the work just given is optional.)

Having gotten the balance into equilibrium by placing weight in notch over point of suspension, take sinker out of water, dry, and immerse in alcohol. It will then be found that the weight and sinker are heavier than the buoyant force of the alcohol. Move weight to next notch toward middle of beam (nine-tenths the distance between central knife-edge and point of suspension of sinker). It will still be too heavy. Move to next notch (eight-tenths the total distance). That will be a trifle too light, showing that the alcohol is something over eight-tenths as heavy as water (over sp. gr. 0.800). Use the two smaller weights—one one-tenth as heavy as the first, the other

one-tenth as heavy as the second. The number of notch on which the second will all but produce equilibrium will give the second decimal point of the specific gravity (thus, if it all but balances on notch No. 2, the specific gravity will be 0.820). Then, with the third weight, bring balance to perfect equilibrium, and use this notch number as the third decimal point place.

Immerse dried sinker into *glycerin*, and weigh as before. It will be found that this liquid will buoy up the beam even when the heaviest weight is placed in the notch directly over the point of suspension of the sinker; that the buoyant force of glycerin is greater than that of water. Therefore, leaving the heaviest weight on the notch over point of suspension of sinker, where it represents the specific gravity 1.000, take the mate to the heaviest weight and place it on the notch where it most nearly balances the beam—if, when on notch three, it is too heavy, then put it on notch two, and if this is a trifle too light, consider the specific gravity as 1.20, and fill out the second and third weights on the appropriate notches as in the alcohol experiment. In this way deduce the specific gravity of glycerin.

Specific Gravity of Solids.—As learned on p. 53, the two important methods of taking the specific gravity of solids are, first, comparing the weight of a solid to the weight of an equal volume of water, and, second, by dividing weight in air by loss of weight of the solid in water. In the latter case we divide by *loss of weight in water* because, according to the Archimedes theorem (p. 54), loss of weight in water equals the weight of water displaced when the body is immersed in water; also equals the weight of a bulk of water equal to bulk of body immersed.

The first part of the following work is given in the hope of proving the Archimedes theorem to the student:

1. Take 20 Gm. brass weight and weigh in air with other weights to prove it is 20 Gm.

Then take test-tube and half fill it with water, marking water-level by a scratch with a file or with a piece of string or rubber band. Then sink therein the 20 Gm. brass weight, to which a silk thread is attached. The water will rise, the amount displaced being, of course, exactly equal to the bulk (the volume) of the weight. Mark point to which water rises as the original level of water was marked. Then remove weight carefully, bring water in test-tube to original level if any is lost in removing weight, and pour in water from graduate to highest mark, and thus measure in cubic centimeters the amount of water displaced.

Since 1 Cc. distilled water weighs 1 Gm. we can put down—
20 Gm. brass weight displaces — Cc. water.

has same volume as — Cc. water.

has same volume as — Gm. water.

Since specific gravity is telling how many times heavier a substance is than the same bulk of water, we can deduce the specific gravity of the brass thus:

Weight of brass Weight of same

(20 Gm.) ÷ volume of water = Sp. gr. of brass.

Now, take same brass weight, hang to the hook from which the scale of the balance is suspended, immerse in water contained in a beaker

held independent of the hanging scale pan, and weigh immersed in water.

As in the experiment with the Mohr's balance, it will be seen that the brass weighs less in water than in air, due to the buoyant force of the water. Weight in air minus weight in water equals *loss of weight in water*.

If the operation is conducted with care, it will be found that the *loss of weight of brass in water is exactly the same as the weight of the water displaced* in the previous experiment, thus proving the Archimedes theorem. Lastly, the loss of weight in water equals the weight of an equal bulk of water, hence, when more convenient, instead of finding the weight of an equal bulk of water by the test-tube experiment, we can weigh the substance in water and deduce specific gravity as follows:

Weight brass in air $\div$ Loss weight brass in water = Sp. gr. of brass.

Specific Gravity of Wax.—To estimate the specific gravity of a body by "loss of weight in water," the substance must be completely immersed. Wax and many other substances float on water, and to get the specific gravity of such a body, it has to be forced beneath the surface of water by means of an appropriate weight, say the 20 Gm. weight of the preceding experiment. The loss in weight of such a sinker under water then enters into the calculation, rendering it somewhat more difficult, as the following data must be taken:

Weight of wax in air.....	_____ Gm.
Weight of 20 Gm. brass weight in air.....	_____ Gm.
Weight of both together in air.....	_____ Gm.
Weight of both together in water.....	_____ Gm.
Loss of weight of both together in water.....	_____ Gm.
Loss of weight of brass alone in water.....	_____ Gm.
Loss of weight of wax alone in water.....	_____ Gm.

Then apply rule:

Weight of wax in air $\div$ Loss of weight of wax in water = Sp. gr. of wax.

Remarks.—In performing this experiment, the wax must be firmly attached to the silk thread by which the brass weight is suspended, and both immersed well under water, as in the foregoing experiment. In all cases of this kind great care must be exercised in removing all air-bubbles from the immersed solids, as such bubbles render the solids more buoyant, and hence vitiate the results.

When wax data are worked out, there will be noticed what seems at first an astonishing fact—that *the wax loses more weight in water than it actually possessed in air*. This can, however, be understood if we remember that weight is a purely relative state; that it represents the downward force of the earth's gravitation exerted on a solid with naught but the air to interpose. On the other hand, when immersed in water, the buoyant force of that liquid—the repelling force of its molecules—pushes the solids *up* the same time that gravitation is drawing it *down*.

In the case of solids lighter than water, the upward force of the water is greater than the downward force of the earth; hence the "loss of weight in water" is greater than "the weight in air."

Specific Gravity of Alum.—Many solids dissolve in water, and these cannot be weighed in water, hence in such cases the specific gravity of such solids must be determined by weighing the solid in some liquid in which it will not dissolve. In this case we employ the oil of turpentine, the specific gravity of which has just been taken with the pycnometer.

The alum suspended by silk thread is weighed in air, then is immersed in a beaker containing oil of turpentine, and weighed, when we get the following data:

Weight of alum in air ——— Gm.

Weight of alum in turpentine ——— Gm.

Loss of weight of alum in turpentine ——— Gm.

If we divide the weight of the alum in air by its loss of weight in turpentine, we find how many times heavier it is than oil of turpentine. This we do not care to know; what we want to know is, how many times heavier it is than water; hence we must indirectly estimate what the loss of weight of the alum would be in water. This we can figure out by the following proportion:

$$\left\{ \begin{array}{l} \text{as the sp. gr.} \\ \text{of turpentine} \end{array} \right\} \text{ is } \left\{ \begin{array}{l} \text{to the sp. gr.} \\ \text{of water} \end{array} \right\} \text{ so } \left\{ \begin{array}{l} \text{the loss of weight} \\ \text{in turpentine} \end{array} \right\} \text{ is } \left\{ \begin{array}{l} \text{to the loss of} \\ \text{weight in water} \end{array} \right\}$$

$$\frac{a}{1.000} : \frac{b}{c} :: \frac{c}{a} \times \frac{b}{c}$$

$$\times = \frac{b \times c}{a}$$

Having thus deduced loss of weight of alum in water, we estimate its specific gravity by the regular rule:

Weight in air of alum $\div$ Loss of weight in water = Sp. gr. of alum.

PHARMACEUTIC MANUFACTURING

MAKE GRANULATED COPPER SULPHATE

Copper sulphate..... 25 Gm.
Water..... 35 Cc.

Put copper sulphate into pint flask, add the water, and dissolve by aid of heat, putting flask over Bunsen burner on wire gauze. When dissolved, filter through plaited filter into pint evaporating dish. Put dish on wire gauze, and evaporate with constant stirring until pellicle forms on surface. Then take from heat and stir constantly until cold. Throw mass on plain filter, and when mother liquor has drained off, open filter and dry granules between sheets of paper.

Notes.—Folding plaited paper see p. 130.

Pellicle.—The “scum” of crystals forming on surface of the evaporating liquid.

Plain Filter.—See page 130.

Wet both filters with water before using.

Avoid formation of white powder (the exsiccated salt) on edges of the liquid.

The *mother liquor*, after filtration, can be thrown away. (See p. 145.) Avoid use of iron spatula in all this work with chemicals. Use glass rod whenever possible.

MAKE GRANULATED ALUM

Alum.....	25 Gm.
Water.....	25 Cc.

Dissolve the alum in the water in pint flask, as directed in granulated copper sulphate, filter into pint evaporating dish, and evaporate on wire gauze with constant stirring until pellicle forms. Collect granules on plain filter—in short, treat exactly as granulated copper sulphate.

Remarks.—The only additional precaution is to avoid evaporating too far, as a sticky mass (partially exsiccated alum) will result.

MAKE GRANULATED POTASSIUM CHLORATE

Dissolve 25 Gm. potassium chlorate in 65 Cc. water in pint flask, filter through plaited filter into pint evaporating dish, and evaporate on *water-bath* until pellicle forms. Stir until cold, and collect granules on plain filter as in granulated copper sulphate.

Remarks.—*Water-bath.*—See p. 75.

Used because the chlorate must not be heated intensely, lest it decomposes; — $\text{KClO}_3 = \text{KCl} + 3\text{O}$.

Note the different solubility of these three salts in hot water

MAKE GRANULATED FERROUS SULPHATE

Copperas.....	25 Gm.
Water.....	25 Cc.
Diluted sulphuric acid.....	i. 25 Cc. (about 20 drops).
Card teeth.....	sufficient quantity.
Alcohol.....	6 Cc.

Place the ferrous sulphate in pint flask, add the water mixed with the acid, and lastly add a pinch of card teeth; warm gently on wire gauze over Bunsen burner until dissolved and the solution is a green color. Then filter through plaited filter into tared evaporating dish, and evaporate on sand-bath until the liquid weighs 38 Gm. Then stir constantly until cool and throw mass on to funnel stopped with plug of absorbent cotton.

When the granules are completely drained of the mother liquor, pour the alcohol on the granules, and when this is drained, throw granules on filter-paper and dry quickly without aid of heat.

Remarks.—This is practically the official process for making Ferri Sulphas Granulatus, U. S. P. (See p. 554.)

Card teeth are pieces of bright iron wire, and a “pinch” means 2 to 5 Gm. When these are added to the solution in the presence of the acid, hydrogen is produced (note evolution), and this reduces any ferric sulphate present in the copperas to ferrous sulphate. This change is easily noted in the conversion of the brownish liquid to a pure green tint.

Tared dish means that the evaporating dish must first be weighed, and it is well to mark with lead-pencil its weight on the outside of the dish. The liquid is then evaporated until dish and contents weigh the weight of the dish plus 38 Gm.

Sand-bath.—Any flat dish containing sand will do. (See p. 73.)

Collection of Granules.—Observe, instead of using a plain filter,

the granules are collected on a plug of cotton in neck of funnel. This is to get the water off as quickly as possible, since green ferrous sulphate is very easily converted (at least partially) into red-brown ferric sulphate, especially when water is present.

Washing with alcohol is done to remove last traces of water, and the granules, when thrown on filter-paper, are wet with alcohol and not with water. Alcohol being more volatile, the granules dry more quickly.

Dry at Low Heat.—No artificial heat must be used, else the green granules will change to a white powder—to the effloresced salt. So soon as dry, the granules should be bottled.

Granular salts are preferable to crystals in prescription work, because more easy to weigh and to dissolve. They usually save the necessity of powdering in a mortar.

MAKE CRYSTALLINED SODIUM SULPHATE

Dissolve 50 Gm. sodium sulphate in 20 Cc. warm water (not boiling) in pint flask, and filter into beaker glass. Let stand, and note sudden formation of crystals with evolution of some heat. Let stand until next day, and then collect crystals on plain filter and dry. The mother liquor which drains off may be evaporated some and may yield another batch of crystals, though not so fine nor so pure as the first.

Remarks.—The sudden formation of crystals occurs only when conditions are just right. (See p. 475.)

Filtration.—In all cases where filtration is for the purpose of clearing a liquid, a plaited filter is used. In case the solid part—be it in the form of granules, crystals, or precipitates—are wanted, plain filters are employed. (See p. 130.) Whenever aqueous liquids are to be filtered, the filter-paper should be first wet with water.

MAKE CRYSTALLINE FERROUS SULPHATE

Put 25 Gm. copperas in pint flask, and add thereto 25 Cc. water and 5 Cc. diluted sulphuric acid and a pinch of card teeth, and dissolve with gentle heat. Filter into beaker, and put in a pinch of card teeth and let stand until good “crop” of crystals are obtained. (Say, one to three days.) Collect these on plain filter and dry.

Remarks.—This process resembles making granulated ferrous sulphate, for remember that granules are merely minute crystals. (See p. 148.)

Card teeth are added to filtered liquid to keep the crystallizing ferrous sulphate from being oxidized to ferric sulphate. They should be carefully removed from the finished crystals.

The *mother liquor* may be concentrated; when more, but poorer, crystals are obtained.

MAKE ROCK CANDY

Dissolve 25 Gm. sugar in 10 Cc. water by means of gentle heat in Erlenmeyer flask; transfer, without filtering, into test-tube, stick a glass rod or piece of twine therein, and let stand several days. The sugar will crystallize on sides of tube and on rod in large crystals.

Remarks.—The sugar solution (syrup) is too thick to filter. Rock candy is made commercially by pouring such a thick syrup into wooden vats containing frames strung with cord, and the sugar in crystallizing deposits on the cords, making the rock candy in strings. The cord acts as a nucleus. (See p. 145.)

MAKE EXSICCATED ALUM, U. S. P.

Recipe.—Alum, in small pieces..... 20 Gm.
To make.. 11 Gm.

Place the alum in a tared shallow porcelain dish so as to form a thin layer, and heat it on a sand-bath until it liquefies. Then continue the application of a moderate heat, with constant stirring, until aqueous vapor ceases to be disengaged, and a dry, white, porous mass weighing 11 Gm. is obtained. When cold, reduce the product to a fine powder, and preserve it in well-stoppered bottles.

Remarks.—Exsiccation is driving off water of crystallization from a crystal, and is here directed to be performed to show the student that a dry crystal does contain water, and that in large amounts.

On heating, the crystal liquefies by dissolving in the crystal water, and on further heating the water can be seen to vaporize, and if wanted, the steam can be condensed on some cold surface. The loss of water is shown in the fact that 20 Gm. of crystal alum yields only 11 Gm. dried alum, representing a loss of 9 Gm. of water, or 45 per cent. $-\frac{9}{20} \times 100 = 45$ per cent.

What is the actual percentage of water in $K_2Al_2(SO_4)_4 + 24H_2O$. Estimation by molecular weight (p. 378).

The dish must be tared and the mass heated until it weighs 11 Gm. plus weight of dish. Be sure to wipe all sand off dish before weighing, and also scrape all adhering alum from stirring rod and put into dish.

Stirring the tough mass is a matter of some difficulty. Best accomplished by holding a glass rod firmly down across top of dish and stirring with a smaller rod.

MAKE EXSICCATED FERROUS SULPHATE. U. S. P.

Recipe.—Ferrous Sulphate, in coarse powder..... 20 Gm.

Allow the salt to effloresce at a temperature of about 40° C. (104° F.), in dry air, and then heat it in a porcelain dish, on a water-bath, constantly stirring, until the product weighs about 13 Gm. Lastly, reduce the residue to a fine powder, and transfer it at once to perfectly dry, well-stoppered bottles.

Remarks.—*Effloresce*, losing water of crystallization at low temperatures (p. 148). Effloresce and finally exsiccate at low temperature, to avoid possible oxidation.

Note change from green crystals to a gray-white powder.

Note loss in weight from 20 Gm. to 13 Gm., a loss of 7 Gm. water, or 35 per cent. $(\frac{7}{20} \times 100 = 35)$.

This is not all the water in the crystals. Estimate by molecular weight percentage of water in $FeSO_4 + 7H_2O$.

SUBLIME BENZOIC ACID

Take benzoin (say, 5 to 10 Gm.), rub with about twice the quantity of sand, and put in tin ointment box. Cover with paper cornucopia, and heat on sand-bath not exceeding 120° C. for an hour. Then open paper and note small shining crystals (Fig. 246).

Remarks.—*Sublimation.* (See p. 105.)

For condensing the vapor of solids, cool air is usually sufficient. Avoid high heat, as it causes decomposition, with formation of very irritating vapors.

SUBLIME CAMPHOR

Take lump of camphor (5 to 10 Gm.) and put in small evaporating dish. Cover same with small inverted funnel, which should fit inside the dish, and place over neck of same the beaker. Place on water-bath and heat. The camphor sublimes, and its vapors condense in funnel and beaker in handsome crystals (Fig. 106).

POWDER FOLDING AND WRAPPING

Camphor..... 5 Gm. (powder with a few drops of alcohol).
Zinc oxide..... 5 Gm.

Triturate together in mortar, divide by eye into 12 papers, fold, put into box, and wrap box, making neat package.

Remarks.—*Powdering camphor* is an example of pulverization by intervention (p. 117).

Folding of Powder Papers.—Crease all papers, lay down evenly, and proceed as on p. 296.

Wrap box in paper neatly, as explained on p. 298.

The division of the powders can be gotten accurately only by weighing each separately. In this case each powder should weigh—

$$\frac{5+5}{12} = \frac{10}{12} \text{ Gm., or } 0.83 \text{ Gm.}$$

The process is not difficult after one becomes expert at it. In this case divide by eye (see p. 294), and then prove the uncertainty of this plan by weighing three finished powders taken at random.

DRUG GRINDING

Reduce gentian root by means of mill or iron mortar to a No. 30 powder, using a No. 30 sieve in the work. Take 50 Gm. of the powder and make into a neat package. (See p. 300.)

MAKE FLUIDEXTRACT OF GENTIAN

Gentian..... 50 Gm.
Diluted alcohol, U. S. P..... q.s.

Put drug into pint evaporating dish, and moisten with 50 Cc. dilute alcohol; put into 8-ounce wide-mouthed bottle, cork, and let stand forty-eight hours. Then percolate.

PERCOLATION

Take percolator, put cork and percolating tubing (p. 161) into neck, and pour water in to see if it leaks. If it does not leak, pour out water, remove the cork and tubing, and wipe percolator dry;

insert plug of cotton in neck of percolator and a layer of cotton on that. Then pour in the moist gentian and pack it firmly with gentle and uniform pressure. Then cut a piece of filter-paper so as to fit inside the percolator, and place this on top of the packed drug, laying a piece of glass—glass stopper—on top to hold the paper down. Then fit the rubber tubing and cork to percolator again, place percolator on ring (p. 157), pour on the drug 100 to 150 Cc. diluted alcohol, and cover top of percolator in some way to prevent evaporation. Place percolator tubing into 8-ounce wide-mouthed bottle, which has been previously graduated to 40 Cc. Let percolation proceed at the rate of 2 to 5 drops a minute, regulating the flow by raising the receiver if it runs too rapidly, or lowering same if it runs too slowly. When 40 Cc. of percolate have dropped through, let the percolate drop into pint flask and cork the wide-mouthed bottle containing first percolate, labeling it "reserved portion." Continue percolating into pint flask until the drug is exhausted (perhaps more than a pint of percolate will be necessary), and when this is accomplished, cork the pint flask and label "weak percolate." It is needless to say that as the first menstruum (the diluted alcohol) passes through the drug, more menstruum is poured on, care being taken always to have some menstruum covering the drug. When the drug is exhausted, the weak percolate is to be distilled.

Remarks.—*Percolation.*—For full description, see p. 155.

Testing Percolator Fittings.—This is essential, for if the tube leaks, the percolation will prove a messy business.

Plug with Cotton.—This can best be done with a long glass rod or tube, though a piece of wire answers. The percolator must be perfectly dry.

Handling the Drug.—*Grind to only No. 30 powder*, because the cells of gentian are large, and most of them are ruptured when in No. 30 powder. (See p. 158.)

Moistened before packing, so as to permit the drug-cells to swell. If put in percolator dry and menstruum then poured on, the drug swells so much that percolation is impeded. (See p. 158.)

Pack Drug Evenly.—If one side is packed more loosely than the other, the menstruum will run down the looser side, and that tightly packed will not be exhausted. Uneven packing is shown in the descent of the first menstruum through the drug. Should pass down in a perfect ring. (See p. 159.)

Percolation.—*Do not pack too tightly*, else the percolate will drop too slowly. Sometimes, under these circumstances, the percolate refuses to drop, and the only thing to do is to empty out drug and pack again. Sometimes the percolate can be "coaxed" into dropping by applying suction at end of tube, or by filling percolator with menstruum. If the first 40 Cc. heavy percolate can be gotten through, the lighter weak percolate will usually run without trouble.

Filter-paper on top, to keep upper layer of powder from floating to top of menstruum. Glass weight holds the filter-paper down.

Reserve First 40 Cc.—If properly percolated, the first 40 Cc. will contain the soluble principles of about 40 Gm. of drug.

Percolate to Exhaustion.—The strength of the remaining 10 Gm. of drug is hard to remove, requiring about ten times the amount of

menstruum as does the reserved portion. Percolate until the drops of liquid are only a faint straw color and almost devoid of bitterness.

Menstruum.—The solvent used in exhausting drug. In this case the menstruum is diluted alcohol, U. S. P.—equal volumes of alcohol and water.

Keep Drug Covered with Menstruum.—If not, the menstruum in the upper layers evaporates, leaving fissures, and when fresh menstruum is poured on, it will run through the fissures rather than through the drug.

Distillation of Weak Percolate.—Fit pint flask about half filled with weak percolate with a cork, bore hole through cork with rat-tail file or with cork-cutter, and in this hole snugly fit a piece of glass tubing bent to an angle of 60 degrees; pass the other end of this tube through hole in a cork, fitting into neck of Liebig condenser.

Immerse flask in water-bath, preventing direct contact with bottom by having rest on wad of paper. Connect flask and condenser, fastening latter in place with appropriate clamps, and pass a stream of water through condenser, entering from the lower end and emerging from top. Get water either from water main or by syphoning from pan of water placed on shelf.

Start heat under water-bath and continue lively heat, replenishing water in bath when necessary. Collect distillate in second pint flask, and continue distillation until over half of the weak percolate has distilled over. Then pour residue in pint evaporating dish, and evaporate on water-bath with constant stirring until residue is of the consistency of soft extract.

Remarks.—Rig up distilling apparatus as on p. 95.

For glass bending and cork boring, see p. 98.

Condensing water run in lower end of condenser—not the upper end, as the heated water rises and must be removed from highest level.

Only about three-fourths of the percolate is distilled, since percolate is only half alcohol, and that is what we wish to save. Since the water is of no value, we evaporate it in open air, as it will evaporate more quickly that way than by distillation.

The alcoholic strength of the distillate can be estimated by taking its specific gravity and referring to alcohol table in the pharmacopœia.

Do not have the distilling flask full of percolate, as it will distil too slowly or froth over into condenser. If you have a pint of percolate or over, add same to distilling flask in portions, pouring in new portions as that part already in flask has evaporated.

Evaporate Residue with Constant Stirring.—Remember that the residue is now aqueous, and that you cannot *boil* water on a water-bath. Remember that evaporation below the boiling-point can be rapidly accomplished only by constant stirring. If the dish is left on water-bath without stirring, the evaporation will be very slow.

Finishing.—The soft extract is dissolved in the reserve portion, and the mixture poured into a graduate. The evaporating dish is washed out with about 5 Cc. diluted alcohol, and this poured into the graduate, and then enough diluted alcohol is added to make finished fluidextract measure 50 Cc. The mixture is thoroughly stirred, and so made constitutes Fluidextract of Gentian, U. S. P.

Remarks.—*The weak percolate is evaporated to soft extract*—most of the water is driven from it—lest that water precipitate some of the principles dissolved in the reserved portion.

The *finished fluidextract* is usually filtered after standing some months. All large manufacturers let their fluidextracts thus “age” for from six months to a year, so that when bottled and shipped, no further precipitation is apt to occur.

MAKE FLUIDEXTRACT GENTIAN BY REPERCOLATION

Repercolation.—In making a fluidextract by repercolation, the drug is to be of the fineness directed by the pharmacopœia, and the menstruum used that first directed by the U. S. P. That is, in some cases the pharmacopœia directs in making a fluidextract by partial evaporation for the reserved portion a menstruum different from that required for the weak percolation; thus in making fluidextract of pomegranate the pharmacopœia directs the use of a menstruum consisting of glycerin, 100 Cc., and diluted alcohol, 900 Cc., at first, and then diluted alcohol for the rest of the process. In making fluidextract of pomegranate by repercolation the first menstruum is used all the way through.

The Process.—Weigh out 50 Gm. of the drug allotted, moisten it with 50 Cc. of the menstruum directed by the U. S. P., and macerate in an 8-ounce wide-mouthed bottle for the time directed by the pharmacopœia. At the end of this time pack in percolator, as directed on p. 1102, and percolate exactly as there described, with one exception. This exception is the caring for the successive portions of the percolate by putting it in separate bottles. Clean and label six bottles. An 8-ounce bottle label “Finished Fluidextract”; four 4-ounce bottles labeled “weak percolate No. 1,” “weak percolate No. 2,” “weak percolate No. 3,” “weak percolate No. 4,” respectively. Lastly, a pint bottle labeled “weak percolate No. 5.”

Collect the percolate in an 8-ounce wide-mouthed bottle, previously measured and marked 40 Cc. and 50 Cc. by scratching with a file. Pour the first 40 Cc. of percolate into “finished fluidextract” bottle, the next 50 Cc. in “weak percolate No. 1” bottle, and the following 50 Cc. portions into “weak percolate Nos. 2, 3, and 4” bottles, respectively. Lastly, percolate the drug to exhaustion, collecting all this percolation into “weak percolate No. 5” bottle.

When “weak percolate No. 1” and “weak percolate No. 2” are collected, a second batch of 50 Gm. of drug is weighed and moistened with “weak percolate No. 1”; macerate it the required time, pack in percolator, and pour on “weak percolate No. 2.” When this has almost all run through, add “weak percolate No. 3,” then “weak percolate No. 4,” then “weak percolate No. 5,” and, lastly, menstruum enough to exhaust.

Collect the percolate in the 8-ounce wide-mouthed receiving bottle and put, first, 50 Cc. of percolate into “finished fluidextract” bottle, the second 50 Cc. into now empty “weak percolate No. 1” bottle, the next into “weak percolate No. 2” bottle, the fourth and fifth 50-Cc. batches into bottles Nos. 3 and 4, and the rest of the percolate to exhaustion in “weak percolate No. 5” bottle. When “weak percolate No. 1” and “weak percolate No. 2” from the second

batch of drug are obtained, weigh out a third batch of 50 Gm. drug and treat it exactly as you did the second batch. When this third batch has been percolated far enough, that is, when weak percolates Nos. 1 and 2 have been obtained from it, get a fourth batch of 50 Gm. drug started, macerating and percolating, making it exactly as you did batches Nos. 2 and 3.

When this fourth batch of drug is exhausted, the operation is at an end, and we will have, as the result, $40+50+50+50$, or 190 Cc. finished fluidextract from the 200 Gm. of drug and weak percolates Nos. 1, 2, 3, 4, and 5.

These weak percolates are to be securely corked and placed in a safe, cool place, there to rest until more of the same fluidextract is needed. Then they are to be taken out, a new batch of 50 Gm. of drug weighed out and macerated and percolated, exactly as before, and the process repeated three times, thus making 200 Cc. finished fluidextract from the 200 Gm. of drug.

MAKE PILLS OF ACETANILIDE

Powdered acetanilide. 1 Gm.

Put on pill-tile, and bring in contact with a spatula containing a sufficient amount of excipient. Knead the excipient into the powder by means of the spatula until a pliable pill mass is obtained. Scrape the mass into one lump, dust the pill-tile with powdered starch, roll the pill mass out with the spatula into a cylinder like a pencil until it is the length of ten divisions on the scale on the tile. Then cut the cylinder with spatula into ten equal parts, and roll each between *clean* thumb and forefinger until perfectly *spheric*. Then put in pill box, dust with starch, wrap box in paper, making a neat package (p. 298).

Remarks.—*Excipient.*—Substance used to make drugs, liquids as well as solids, into plastic mass (p. 314).

A *good excipient* for powders is glycerite of tragacanth:

Powdered tragacanth.	3 drachms
Triturate with glycerin.	3 fluidrachms
Then add water.	$\frac{1}{2}$ fluidounce.

Transfer jelly into appropriate jars.

The *amount of excipient required* can be learned only by experience. Roughly speaking, in above case a lump about the size of a pea should suffice. To make a good mass the entire amount of excipient required should be added at one time.

If mass is *too soft*, add another 1 Gm. powdered acetanilid and make 20 pills.

Masses are made on tiles only when small amounts of pills are to be made, and when there is no difficulty in forming the mass.

Pliable Pill Mass.—See p. 314.

Cutting pills on tile with spatula gives irregular results. Every drug-store should have a pill-cutter. (See p. 316.)

Pills are dusted to prevent their sticking together. White pills should be dusted with powdered starch; dark pills, with lycopodium.

MAKE COMPOUND CATHARTIC PILLS, U. S. P.

<i>Recipe.</i> —Compound extract of colocynth.....	4.00 Gm.
Mild mercurous chloride.....	3.00 Gm.
Resin of jalap, in fine powder.....	1.00 Gm.
Gamboge, in fine powder.....	0.75 Gm.
Diluted alcohol, a sufficient quantity,	

To make.. 25 pills.

Mix the powders intimately, then incorporate a sufficient quantity of diluted alcohol to form a mass, and divide it into 25 pills.

Remarks.—In *tritulating powders* always first rub up the ingredients prescribed in smallest amount with a small quantity of the other substances, and add the rest of the drugs gradually with constant stirring—this to insure thorough blending of the most potent drugs.

The *excipient* in this case is diluted alcohol, the compound extract of colocynth containing soap and aloes, which combine with the water to make a plastic mass. Five drops should suffice, provided enough “elbow grease” is used. In fact, if the mortar is warmed and the pestle is handled vigorously, the mass can be formed without use of diluted alcohol.

Working up the mass in a mortar with a pestle is a process hard to explain by words. The pestle is firmly grasped in the right hand with the end of the handle resting against the center of the palm, while the mortar is held fast with the left hand. The mass is worked by pressing down on it with the pestle with a rotary movement to the right. From time to time the mass is carefully scraped from the pestle and from the sides of the mortar and worked again, the process being continued until a thoroughly plastic mass results.

Cutting the cylinder into pills must be done indirectly when the scale on the pill-tiler is for ten or twenty pills, if twenty-five pills are directed. If the scale has twenty markings, roll the cylinder out to twenty and cut into five equal lengths (four spaces each). Then take each length and roll until it is five spaces long, and cut into five pills. Treating the other four lengths similarly, we have in the end twenty-five pills. It is needless to say that great care should be taken to have the cylinder of exactly the same thickness through its entire length.

Roll pills between first finger and thumb as in making pills of acetanilide.

Dust pills with lycopodium.

MAKE PILLS OF PHOSPHORUS, U. S. P.

<i>Recipe.</i> —Phosphorus.....	0.015 Gm.
Althæa, in No. 60 powder.....	1.500 Gm.
Acacia, in fine powder.....	0.750 Gm.
Chloroform,	
Glycerin,	
Water,	
Balsam of Tolu,	
Ether, each, a sufficient quantity,	

To make.. 25 pills.

Dissolve the phosphorus, in a test-tube, in $1\frac{1}{4}$ Cc. of chloroform, with the aid of a very gentle heat, replacing from time to time any of the chloroform which may be lost by evaporation. Mix the althæa

and acacia in a mortar, next add the solution of phosphorus, then immediately afterward a sufficient quantity (about 1 Cc.) of a mixture of two volumes of glycerin and one volume of water, and quickly form a mass; divide it into 25 pills.

Dissolve 10 Gm. balsam of Tolu in 15 Cc. of ether, shake the pills with a sufficient quantity of this solution until they are uniformly coated, and put them on a plate to dry, occasionally rolling them about until the drying is completed.

Keep the pills in a well-stoppered bottle.

Remarks.—*Handling the phosphorus* must be done with care, as it is very unstable, igniting spontaneously when exposed too long to the air. Phosphorus should always be weighed *under water*, that is, a beaker or other vessel containing water should be counterpoised on the scale, and the phosphorus immersed in the water while weighing. In the same way the large piece of phosphorus should be cut while under water contained in pint evaporating dish.

The *cutting of phosphorus* is easy, since it is a soft, waxy solid, readily sliced with an ordinary knife. Always cut off the outer oxidized layer, and use the fresh phosphorus from the middle of the stick.

The *solution of phosphorus* is made by quickly freeing the weighed phosphorus from adhering water by pressing between filter-paper and dropping the lump into the chloroform contained in a test-tube before the phosphorus has time to ignite. If the phosphorus dissolves slowly in the chloroform, the solution can be aided by the use of gentle heat, adding extra chloroform to make up for loss of evaporation.

The *mass must be worked up* as rapidly as possible lest it *catch fire*. If it does ignite, carry to sink and turn on the water from the faucet.

Use enough excipient to make a rather soft mass, for a firm mass becomes rubber-like on rolling out. Ten to twelve drops usually suffice.

In cutting the pill cylinder use the same plan as with compound cathartic pills if the pill-tille scale has not twenty-five spaces. Use lycopodium as dusting-powder.

Coating the pills does not yield handsome results; in fact, no pill coating can be well done without special appliances. (See p. 318.) In this special case the coating is not put on to enhance the appearance of the pill, but to protect it from atmospheric action (p. 324). In coating, put pills in small dish, pour on ten to twenty drops of the tolu solution, and rotate until dry. Then, if necessary, add more tolu solution and rotate further.

PUT UP / CONSEALS

Pulverized sugar. 6 Gm.

Put up in six / conseals (No. 1 size) by directions found on conseal apparatus. (See p. 307.)

MAKE LOZENGES OF POTASSIUM CHLORATE, U. S. P.

Recipe.—Potassium chlorate, in fine powder. 1.8 Gm.
 Sugar, in fine powder. 7.2 Gm.
 Tragacanth, in fine powder. 0.36 Gm.
 Water, a sufficient quantity,

To make. . 12 troches.

Mix the sugar with the tragacanth by trituration in a mortar; then transfer the mixture to a sheet of paper, and by means of a bone spatula mix with it the potassium chlorate, being careful, by avoiding trituration or pressure, to prevent the mixture from igniting or exploding. Lastly, with water, form a mass to be divided into twelve troches.

Remarks.—*Beware of trituration of dry potassium chlorate with sugar or other oxidizable substances, as a frightful explosion is apt to occur.* Hence the pharmacopœia directs mixing these ingredients on paper with bone spatula. A metallic spatula can be used, however, if one is cautious in the manipulation.

Working the powders into a mass can be safely done with mortar and pestle, provided the mass is well moistened. Do not use too much water, however, lest a very soft mass results—1 Cc. should suffice.

Roll out the mass as if it were biscuit dough, but do not roll too thin. In making twelve lozenges the mass is generally rolled out broad enough comfortably to cut eight lozenges. The scraps are then worked up and rolled out so that three lozenges can be cut, and the scraps now remaining should be just enough to make the twelfth lozenge—no more, no less.

Both pills and lozenges should be air-dried before putting away. If bottled while moist, they will grow moldy.

MAKE BELLADONNA SUPPOSITORIES

Extract of belladonna.....	0.32
Oil of theobroma.....	10.00.

Make five suppositories.

Weigh the extract out on a piece of paper and transfer by means of a spatula to the point of the pestle, and make into paste with about five drops water in a warm mortar.

In the meanwhile melt the oil of theobroma in small evaporating dish on water-bath, and when completely fused, pour about one-tenth of the oil into warm mortar containing the extract. Triturate until a complete blending of the oil and the extract takes place, and then transfer the still liquid mass into the rest of the oil of theobroma in the dish, carefully scraping all from mortar and pestle by means of spatula.

Mix the extract thoroughly with the molten oil of theobroma by stirring the glass rod, and remove from water-bath, and when the first signs of congealing show, pour the mixture into the thoroughly chilled molds, keeping the extract suspended by constant stirring while pouring into mold. Then let mold stand quietly on the ice until solidification is complete, after which remove the suppositories from the mold.

Remarks.—The *extract* used in suppositories should be soft extract, not the powdered form.

Fusing the oil of theobroma should be at a low temperature. Heating too high gives the mass a fried smell.

Do not pour into molds while the mass is hot. Just before it congeals is the proper time.

Stir mass while pouring, else the extract will not be uniformly distributed.

Have molds on ice at least a half-hour before pouring mass in. To get good results, the molds must be thoroughly chilled.

Have interior of molds perfectly dry and clean when mass is poured in. If such is the case, there is no need of using lycopodium to dust inside of mold.

The mass, *on congealing*, if molds are well chilled, contracts and loosens from molds with smooth, shining surface. In this case *suppositories can be removed from mold* by gentle tapping. If they stick, slight downward pressure from the top usually loosens them. If they stick more tenaciously, the molds must be cautiously opened and the suppositories removed by pressing from tip upward.

If the suppositories stick to mold, it is due either to insufficient chilling of molds before melted mass is poured in or to dirtiness of molds.

Most suppository molds hold 20 grains of oil of theobroma. This must be considered in making suppositories, and the amount of oil of theobroma added in making the mass should be just enough to fill the molds.

MAKE SUPPOSITORIES BY HAND

Oil of theobroma.....	8 Gm.
Castor oil.....	q.s.

Grate or slice the oil of theobroma in thin shavings and put into mortar; add about five drops of alcohol and thus pulverize by intervention. Then add five drops castor oil and work into mass as if making pills. Place finished mass on pill-tile, dust with lycopodium, and roll out to mark 8 on scale on pill-tile. Cut the cylinder in half and shape each half into a double cone by rolling gently with spatula. Then cut each double cone in half, and we have *four* suppositories.

Remarks.—This method does not yield as handsome a product as those made by fusion, but it has the advantage of being applicable in all cases.

Avoid contact of the mass with the hand, as the oil of theobroma melts at temperature of the body.

In shaping the suppository, if found more convenient, the mass can be directly divided into four parts by measuring on pill-tile or by weighing, and each part made into a cone.

These pure butter of cocoa suppositories are given merely for practice. Of course, in actual business, they will be directed blended with some medicinal constituents.

MAKE COMPRESSED SUPPOSITORIES

Take oil of theobroma q. s. (say, 10 Gm.). Powder with alcohol, put into suppository machine, and press into both the rectal and the bougie mold. For picture of mold, see p. 338.

Remarks.—Note what is said just above as to fact that in actual business the oil of theobroma is always prescribed combined with some medicinal constituent.

MAKE GLYCERIN SUPPOSITORIES, U. S. P.

<i>Recipe.</i> —Glycerin.....	6.00 Gm.
Monohydrated sodium carbonate.....	0.10 Gm.
Stearic acid.....	0.40 Gm.
Water.....	1.00 Cc.
To make.. 2 suppositories.	

Dissolve the monohydrated sodium carbonate in the water and add it to the glycerin, contained in a dish, on a water-bath; add the stearic acid, and heat the mixture carefully until carbon dioxide ceases to be evolved and the liquid is clear. Then pour the melted mass into suitable molds, remove the suppositories when they are completely cold, and preserve them in tightly stoppered glass vessels.

Remarks.—The manufacture of these suppositories involves a chemical action, the sodium carbonate reacting with the stearic acid to form sodium stearate, and with this the glycerin blends, thereby producing a solid suppository containing about six-sevenths its weight of glycerin.

Weigh the glycerin in small evaporating dish, and to it add the sodium carbonate and later the stearic acid.

Be sure the sodium carbonate is crystalline and monohydrated. The efflorescent salt works poorly.

Shave the stearic acid into thin pieces with knife or spatula before putting into glycerin.

Heat Carefully.—Experience shows, however, at this stage, that the heat of a water-bath scarcely suffices, so when the sodium carbonate is dissolved, remove from water-bath to wire gauze, dry the bottom of dish, and heat *very cautiously* until reaction ceases. *Until escape of carbonic acid gas has ceased.*

Sodium carbonate plus stearic acid or any other acid gives off CO₂ gas, and forms a salt of the added acid.

Stir very slightly in making, and that only with glass rod, merely enough to aid the CO₂ in escaping. If stirred vigorously, the mass will become mixed with air and will not be clear.

The mass *must be clear* when poured into molds, else the finished suppositories will be opaque.

Pour into Suitable Molds.—Note that the finished suppository will weigh over 3 Gm., about twice the ordinary rectal suppository. Hence the ordinary mold will not do. For practice, however, the mass may be poured into the smaller mold, thus making a suppository half the official size. In this case, as in belladonna suppositories, the mold must be thoroughly chilled by placing on ice, and the mass must be almost ready to congeal when poured in.

Removing these suppositories from the mold by tapping will not work; the molds must be opened and the suppositories removed by pressing from tip upward.

MAKE COLD CREAM, U. S. P.

<i>Recipe.</i> —Spermaceti	6.25 Gm.
White wax.....	6.00 Gm.
Expressed oil of almond.....	28.00 Gm.
Sodium borate, in fine powder.....	0.25 Gm.
Stronger rose water.....	9.50 Gm.
To make about.. 50 Gm.	

Reduce the spermaceti and the white wax to fine shavings, and melt them at a moderate heat; add the expressed oil of almond and stir, continuing the heat until the mixture is uniform; then gradually add the stronger rose water, previously warmed, and in which the sodium borate has been dissolved, stirring the mixture rapidly and continuously until it congeals and becomes of uniform consistence.

When this ointment is to be used as a vehicle for metallic salts, the sodium borate should be omitted.

Remarks.—*Melt the spermaceti and white wax* in pint evaporating dish, and to it add the oil of almond.

Dissolve the borax in the rose water in Erlenmeyer flask, and this solution should be almost as warm as the mixed fats. If it is cold, the spermaceti will separate as if curdled.

Do not warm the rose water enough to drive off its odor, however. The finished cold cream is not highly perfumed. If more odor is desired, add—

Oil rose.....	10 drops	} to each pound.
Oil lemon.....	6 drops	
Oil bergamot.....	4 drops	

The oils should be added, not alcoholic solutions of them.

This cold cream turns rancid quickly, and should, therefore, be sold only when fresh. For a permanent cold cream white paraffin oil is substituted for oil of almond. *This substitute must not, however, be dispensed as official cold cream.*

MAKE BLUE MASS, U. S. P.

<i>Recipe.</i> —Mercury.....	3.3 Gm.
Glycyrrhiza, in No. 60 powder.....	1.0 Gm.
Althæa, in No. 60 powder.....	1.5 Gm.
Glycerin.....	0.9 Gm.
Honey of rose.....	3.3 Gm.
To make.. 10 Gm.	

Triturate the mercury with the honey of rose until it is extinguished, and globules of mercury are no longer visible under a lens magnifying at least ten diameters. Add the glycerin, then the glycyrrhiza and althæa gradually, and continue the trituration until the mass is homogeneous. Keep the product in well-closed containers.

Remarks.—*Triturate the mercury with honey of rose and glycerin* in a mortar until extinguished—that is, until no mercury globules are seen and a thin gray paste results. If trituration is stopped, however, the mercury will run together.

Add the powdered drugs and then triturate vigorously. This has the effect of coating each globule of mercury with the powder, thus preventing one from running into the other.

Triturate until no globules are seen under glass magnifying ten diameters—that is, making things look ten times their real size. To accomplish this usually takes a half hour's trituration.

MAKE EMULSION OF COD LIVER OIL
(by Continental Method)

Cod liver oil.....	30 Cc.
Water.....	15 Cc.
Powdered acacia.....	7.5 Gm.
Water, syrup, and flavor, to make.....	60 Cc.

Put the acacia in a dry mortar and add all the oil, triturating the mixture well. Then add the water (15 Cc.) all at once and triturate vigorously until a creamy emulsion results. Lastly, add syrup, 5 Cc., flavor to taste, and add water to make 60 Cc.

Remarks.—The theory of emulsions is similar to that of making blue mass and other preparations of free mercury, the oil being broken into fine globules by trituration and each globule immediately coated with a layer of gummy substance, thus preventing adjacent globules from running together.

The two methods of emulsification are called the English and the Continental processes (see p. 266), and the above recipe is by the Continental process, and practically that recognized by the present pharmacopœia (p. 271).

Note the proportions essential to a successful Continental emulsion: 4, 2, and 1; 4 parts of oil, 2 parts of water, and 1 part of gum.

For flavor, the pharmacopœia directs $\frac{1}{10}$ of 1 per cent. of oil of gaultheria, or an appropriate quantity of oil of bitter almonds (p. 271). The finished acacia emulsion is almost too thick for practical use and contains too large amount of acacia.

The *English emulsion* can be made from less gum, the sole advantage the process possesses.

When oil, gum, and water are triturated a few minutes, a white emulsion should result, the desired effect being indicated by a characteristic crackling sound as the mixture is triturated. If this crackling sound is not heard, and if the mixture has a greasy, "pearly" appearance, it means that the oil is not emulsified—that the emulsion is "split."

Do not Throw Away a Split Emulsion.—Make up another batch as a nucleus, and gradually incorporate the split emulsion, when the entire quantity will be emulsified. There is no better emulsifier than a good emulsion.

MAKE FLASK EMULSION OF COD LIVER OIL

Powdered tragacanth.....	1 Gm.
Gelatin, in No. 80 powder.....	1 Gm.
Cod liver oil.....	20 Cc.
Water, flavor, etc., to make.....	40 Cc.

Put the powders in a dry Erlenmeyer flask, add the oil and shake, then add 15 Cc. water, and shake when an emulsion results.

Remarks.—This formula was devised to replace a class of flask emulsifiers introduced a few years since, and containing the two questionable substances—saponin and saccharin.

It is given merely as an illustration of this kind of work and is not recommended. To make a good emulsion one should be willing to dirty a mortar.

It also shows that almost any gummy substance can be used as an emulsifier.

MAKE EGG EMULSION OF COD LIVER OIL

Glycerite of yolk of egg (U. S. P. 1890).....	10 Cc.
Cod liver oil.....	30 Cc.
Syrup.....	6 Cc.
Brandy.....	10 Cc.
Water.....	4 Cc.

Pour the glycerite into mortar, and add the oil little by little, with constant trituration until completely emulsified. Then add the other ingredients in the order named.

Remarks.—This preparation is emulsified by use of yolk of egg, an emulsion of egg fat with the albuminoids. It is an illustration of the dictum, “the best emulsifier is a perfect emulsion.”

Be careful to add oil gradually, else the emulsion may split. The addition of the last portions of oil may make the product almost too stiff to work, in which event add a few drops of water, say, 1 Cc. *In laboratory work* omit the brandy from this recipe.

The *best flavor for this* is a compound tincture of cardamom *not* colored with cochineal. One-half Cc. suffices with the above quantity.

MAKE EMULSION OF TURPENTINE

Powdered tragacanth..... 2 Gm.

Put in dry 8-ounce wide-mouthed bottle, corked and add—

Oil of turpentine..... 16 Cc.
Then add water..... 30 Cc. and shake.

The finished emulsion can be diluted with water and flavored to any strength directed by the physician.

Remarks.—This is not the recipe provided by the present pharmacopœia for official emulsion of oil of turpentine (p. 271), in which acacia is used as the emulsifying agent, and expressed oil of almond used to make a more stable emulsion.

CHAPTER LX

CHEMICAL PREPARATIONS

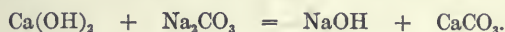
MAKE SOLUTION OF SODA, U. S. P. 1890

Recipe.—Sodium carbonate..... 42.5 Gm.
Lime..... 12.5 Gm.
Distilled water, a sufficient quantity.

Dissolve the sodium carbonate in 100 Cc. of boiling distilled water. Slake the lime with about $7\frac{1}{2}$ Cc. of distilled water, then mix it well with 100 Cc. of distilled water, pour the mixture into a tared flask, and, having heated it to boiling, gradually add to it the solution of sodium carbonate and boil during ten minutes. Then add enough distilled water to the flask to make the contents weigh 250 Gm., and set the flask aside, well stoppered, until the contents are cold. Lastly,

strain the liquid through linen, set it aside in a well-stoppered bottle until it has become clear by subsidence, and separate the clear solution by decantation or by means of a siphon.

Explanation.—Monohydrated sodium carbonate, U. S. P. 1900, 18.4 Gm. can be used instead of the above amount of crystalline sodium carbonate. The chemical reaction in this process is as follows:



The result of the reaction is a liquid containing sodium hydroxide in solution, and a sediment consisting of calcium carbonate (precipitated chalk).

The equation given above is purposely left incomplete. Complete this so that the amount of each element on the two sides of the sign of equality is the same.

Again, the equation does not represent the truth as far as the constituents directed by the U. S. P. is concerned. Lime is not calcium hydroxide, but is calcium oxide, the hydroxide being produced only after shaking the lime, the reaction being $\text{CaO} + \text{H}_2\text{O} = \text{Ca(OH)}_2$.

Again, the sodium carbonate directed above is not the exsiccated Na_2CO_3 , but the crystalline salt containing ten molecules of water of crystallization. So the real reaction runs:



Be sure to complete this equation before proceeding further. (See p. 421.)

Calculations.—Figure out molecular weight of the five substances in above equation, viz.:

Ca atomic weight	=	—	Ca	=	1 × — = —.
O atomic weight	=	—	O	=	1 × — = —.
			CaO molecular weight	=	—.

H atomic weight	=	—	H ₂	=	2 × — = —.
O atomic weight	=	—	O	=	1 × — = —.
			H ₂ O molecular weight	=	—.

Na atomic weight	=	—	Na ₂	=	2 × — = —.
C atomic weight	=	—	C	=	1 × — = —.
O atomic weight	=	—	O ₃	=	3 × — = —.
H ₂ O molecular weight	=	—	10H ₂ O	=	10 × — = —.
			Na ₂ CO ₃ ·10H ₂ O molecular weight	=	—.

Na atomic weight	=	—	Na	=	1 × — = —.
O atomic weight	=	—	O	=	1 × — = —.
H atomic weight	=	—	H	=	1 × — = —.
			NaOH molecular weight	=	—.

NaOH molecular weight = —. The actual weight of NaOH produced from above equation is this molecular weight *times* the number of molecules produced.

Ca atomic weight = —
 C atomic weight = —
 O atomic weight = —

Ca = $1 \times$ — = —
 C = $1 \times$ — = —
 O₃ = $3 \times$ — = —
 CaCO₃ molecular weight = —

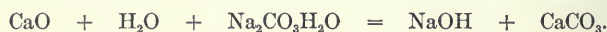
Now write the equation last given with the correct number of molecules, and place under each substance the figures expressing the molecular weights, or, in case more than a molecule of a substance is involved, the combining weights. Then reckon out the following problems:

1. How many grammes absolute sodium hydroxide in 250 Gm. of 5 per cent. solution of soda?

2. How many grammes of lime (CaO) and of crystallized sodium carbonate (Na₂CO₃+10H₂O) needed to make 80 Gm. absolute soda (NaOH)?

3. How many grammes of lime and of crystalline sodium carbonate needed to make 12.5 Gm. absolute soda?

If monohydrated sodium carbonate is used, the equation will run:



(Complete this equation.)

The molecular weight calculation, as far as the sodium carbonate is concerned, will be:

Na atomic weight	—	Na ₂	= $2 \times$ — = —.
C atomic weight	—	C	= $1 \times$ — = —.
O atomic weight	—	O ₃	= $3 \times$ — = —.
H ₂ O molecular weight	—	H ₂ O	= $1 \times$ — = —.
		Na ₂ CO ₃ H ₂ O molecular weight	= —.

For table of atomic weights, see p. 367.

For complete figuring of molecular weights, see p. 363.

For details of balancing equations, see p. 421.

Comparing the figures obtained with the quantities given in the U. S. P. process, a marked discrepancy (increase) in the case of the lime and a slight discrepancy in the case of the sodium carbonate will be noted.

This is due, as far as lime is concerned, to the fact that commercial lime is quite variable in quality, and a large excess will work no harm, inasmuch as it is very sparingly soluble in water. As to difference in amount of sodium carbonate, note that the official process does not yield 250 Gm. of solution of soda; but 250 Gm. of a mixture of it with the precipitated chalk.

Remarks on Process.—*Dissolve the sodium carbonate* in pint flask, and when solution is complete, put into an 8-ounce wide mouthed bottle. Filter only if necessary—if containing dirt.

Be sure that the sodium carbonate is not effloresced. For this reason the non-efflorescent monohydrated sodium carbonate is better.

Slake the lime by placing a lump in a mortar and adding the water, drop by drop, so it can be absorbed by the lime.

Better do this before dissolving the sodium carbonate, as with the small amount of lime used the slaking will take some time. Do not

expect the generation of a large amount of heat in slaking the minute quantity of lime. Heat is developed, but in scarcely appreciable quantity. Slaking of the lime may be hastened by warming mortar on radiator.

When the lime is slaked, it will crumble to a powder. This takes, with the small amount of lime employed, fifteen minutes to a half-hour. When lime is slaked, *mix it well with water*. That is, triturate with, say, 75 Cc. of the water, making a milky paste (milk of lime); pour this into flask, and wash mortar and pestle with the other 25 Cc. water, pouring washings into flask containing the milk of lime.

Be sure to tare flask before pouring in the milk of lime. The rest of the official directions need no special remarks, except the statement that—

The finished product is not to be filtered: at least, through paper, as the alkali will eat the paper. Asbestos wool can be used, however, a bit of this inserted into funnel as if it were a plug of cotton. The pharmacopœia, however, directs decantation.

The precipitated chalk is to be thrown away, as it cannot be freed from the alkali except by extraordinary effort.

MAKE SOLUTION OF CHLORINATED SODA, U. S. P.

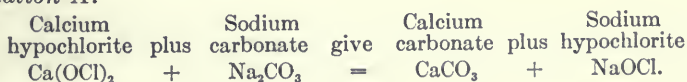
Recipe.—Monohydrated sodium carbonate..... 6.5 Gm.
Chlorinated lime..... 9.0 Gm.
Water, a sufficient quantity,

To make... 100 Gm.

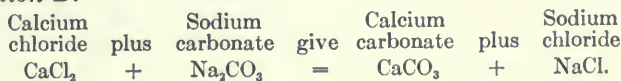
Triturate the chlorinated lime (which should contain not less than 30 per cent. of available chlorine) with 20 Cc. of water, gradually added, until a uniform mixture results. Allow the heavier particles to subside, and transfer the thinner, supernatant portion to a filter. Then triturate the residue again with 20 Cc. of water, transfer the whole to the filter, and, when the liquid has drained off, wash the filter and contents with 10 Cc. of water. Dissolve the monohydrated sodium carbonate in 30 Cc. of hot water, and add this solution to the previously obtained filtrate contained in a suitable vessel. Stir or shake the mixture thoroughly, and, if it should become gelatinous, warm the vessel very gently until the precipitate subsides. Then transfer the mixture to a new filter, and, when no more liquid drains from it, wash the filter and contents with enough water to make the product weigh 100 Gm. Keep the solution in well-stoppered bottles, protected from light, and in a cool place.

Explanation.—The chemical reaction involved in the above process runs in two equations, since the chlorinated lime employed is a mixture of calcium hypochlorite and calcium chloride:

Equation A:



Equation B:



The result of the reaction is a liquid containing sodium hypochlorite and sodium chloride in solution and calcium carbonate (precipitated chalk) as a sediment.

The equations given above are purposely left incomplete. Complete these, so that the amount of each element on the two sides of the sign of equality is the same.

Calculations.—Chlorinated lime, being of variable quality, the molecular quantities cannot be conveniently worked out.

However, figure out:

How many grammes of chlorine in 9 Gm. of 30 per cent. chlorinated lime?

How many grammes of chlorine in 100 Gm. of 2.4 per cent. solution of chlorinated soda?

Comparison of these figures will show that the pharmacopœial process anticipates the conversion of the chlorinated lime into solution of chlorinated soda with the loss of but 10 per cent. of the volatile chlorine—a thing that is theoretically possible, but rarely practically attained.

Remarks on Process.—*Put chlorinated lime in mortar* and triturate with the water.

The *thinner supernatant portion* (see p. 139) contains the soluble part of the chlorinated lime, and this is passed through the filter first. This is an illustration of decantation.

The *heavier particles* are triturated with more water, adding the water gradually with firm pressure of the pestle in order to reduce particles to a pasty condition.

Pour the entire paste on the filter, and thus dissolve the soluble portion left in the chlorinated lime after the first washing.

Use the last 10 Cc. water to wash out mortar and to extract the insoluble part of the chlorinated lime contained on the filter.

Dissolve the sodium carbonate in the water in pint flask, and to this add the chlorinated lime filtrate.

If gelatinous, warm, but that only slightly, lest a large amount of chlorine be driven off.

Filter mixture into a tared 8-ounce wide-mouthed bottle. Both solution of chlorinated soda and also chlorinated lime contain chlorine in a form so loosely combined that the gas easily escapes. Hence in the entire process care should be taken to avoid undue exposure, either to the air or to heat.

MAKE AMMONIUM SULPHATE

Ammonia water (10 per cent.)..... 340 grains or — Gm.
Diluted sulphuric acid (10 per cent.)..... 980 grains or — Gm.

Place the ammonia water in a pint evaporating dish, add the sulphuric acid gradually until faintly acid to litmus-paper, using more than the amount given above if necessary.

Then add extra ammonia water, drop by drop, until alkaline to litmus-paper. Place dish on wire gauze and evaporate to dryness.

Explanation.—This process gives us an illustration of a simple reaction, viz.: ammonia plus sulphuric acid gives ammonium sulphate.

Express this reaction by equation, writing the formula of each of the compounds mentioned above and the number of molecules of each substance entering the equation.

Calculations.—Next work out molecular weight of each of the above compounds (as shown in solution of soda), and through these molecular weights answer the following problems:

1. How many grammes ammonia gas and absolute sulphuric acid are needed to make 132 grains of ammonium sulphate?

2. How many grains of 10 per cent. ammonia water and of 10 per cent. sulphuric acid are needed to make 132 grains of ammonium sulphate?

Remarks on Process.—Convert grains into grammes by dividing by 15.432.

Add the acid gradually to the ammonia, as the reaction is apt to be quite violent

Litmus-paper (see p. 373).

In testing with litmus-paper dip a glass rod into the liquid and thus bring one drop of the liquid on the piece of litmus-paper moistened with water. Never dip the litmus-paper into the mixture.

Add acid until faintly acid—that is, it may chance that the theoretic quantities given above may not yield exact results, due to some variation in strength of either acid or alkali. In such cases we therefore look for results rather than figures, and use an extra quantity of the ingredients to get the results, if necessary.

Thus, if after adding the directed quantity of acid, the mixture is still alkaline (until a drop turns red litmus blue), add acid until faintly acid (turns blue litmus red).

Usually, after the acid is added, the reaction will be distinctly acid, as the ammonia is more apt to lose strength on standing than is the acid. If such is the case, add ammonia until the mixture is distinctly alkaline—until a drop turns red litmus-paper blue.

Be sure to have evaporating mixture alkaline. An excess of acid will spoil the product; an excess of alkali will have no hurtful result, since the excess of ammonia will be driven off as the salt reaches dryness.

Evaporate to dryness, but do not continue heat after dryness—as the ammonium sulphate is volatile.

MAKE AMMONIUM IODIDE

Potassium iodide.....	$\frac{1}{2}$ troy ounce or	— Gm.
Ammonium sulphate.....	109 grains or	— Gm.
Water.....	$\frac{1}{4}$ fluidounce or	— Cc.

Put salts in Erlenmeyer flask, add the water while boiling, stir well, and let cool. Then add 1 drachm alcohol, cool the mixture with ice or running water, and throw the mixture on funnel plugged with cotton. When the liquid has filtered through, wash precipitate with 1 drachm of a mixture, consisting of alcohol, 1 part, water, 2 parts. Then put filtrate in pint evaporating dish, and evaporate solution rapidly to dryness, stirring constantly.

Explanation.—In this process a double decomposition occurs between the potassium iodide and ammonium sulphate, whereby potassium sulphate and ammonium iodide are formed.

Equation:



Complete this equation.

Calculation.—Figure out molecular weights of each of the above substances, and from the molecular weights learn the weights by which the two chemicals combine. Then solve the following problems:

1. How much potassium iodide and ammonium sulphate are needed to make 290 grains of ammonium iodide?

2. How much potassium iodide and ammonium sulphate are needed to make 218 grains of ammonium iodide?

Compare these figures with above recipe, and see how closely they agree.

Remarks on Process.—Looking at the explanation of the chemistry of the process, we see that the products of the reaction between potassium iodide and ammonium sulphate are potassium sulphate and ammonium iodide. Now it chanced that both of these chemicals, like the substances from which they are made, are soluble in water. How are we to obtain the ammonium iodide we desire free from the potassium sulphate?

In all manufacturing chemistry effort is made to get all the *reaction* products save the one we desire in some form different from the one we want.

Thus, in making solution of soda, we choose as the sodium ingredient sodium carbonate, knowing that that would produce with the lime insoluble calcium carbonate, thus leaving the soda itself in the solution, free from any other substance. Again, if we want an insoluble body, say, precipitated chalk, we try to get the other reaction products in a soluble form. (See p. 493.)

In the ammonium iodide we cannot think of any salt we can use that will obtain for us the second reaction product in insoluble form. Every combination we think of will yield some chemical soluble in water, even as is ammonium iodide. Therefore we have to devise some way of separating the iodide from the potassium sulphate, and we accomplish this by use of alcohol, in which the iodide is freely soluble, while the sulphate dissolves but slightly.

Filter through funnel plugged with cotton, even as you did granulated ferrous sulphate. (See p. 1099.)

Evaporate rapidly, as the product is quite unstable, easily giving up its iodide and becoming brown. For this reason, during evaporation, it is advisable to add a drop of ammonium sulphide test solution, from time to time, to prevent decomposition.

MAKE PRECIPITATED CHALK

Marble..... 10 Gm.

Put in beaker and add a mixture of—

Hydrochloric acid..... 23 Gm.
Water..... 45 Cc.

Watch effervescence, and when solution is complete, filter into another beaker. Then add a solution of:

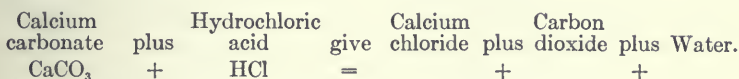
Monohydrated sodium carbonate.....	12.4 Gm.
Water.....	90 Cc.

Stir, let precipitate subside, then decant supernatant liquid, add more water, and finally precipitate on to a plain filter and continue washing until wash liquor no longer gives precipitate with either calcium chloride solution or silver nitrate solution. Then let precipitate dry.

Explanation.—This process is in two stages.

First, the insoluble marble (crude calcium carbonate) is dissolved in hydrochloric acid, forming soluble calcium chloride with the evolution of carbon dioxide, viz.:

Equation A:

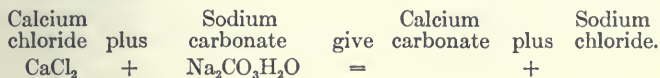


Complete this equation.

The dissolving of marble by hydrochloric acid is for the purpose of purification, it being easier to purify substances when in solution than in solid form. The solution of calcium chloride is then treated with sodium carbonate, when calcium carbonate (precipitated chalk) separates out, while sodium chloride goes into solution.

The reaction is:

Equation B:



Complete this equation.

Calculations.—First, the manufacture of calcium chloride. Work out the molecular weights of calcium carbonate, hydrochloric acid, and calcium chloride, and place under equation given above. Then solve the following problems:

1. How many grammes of absolute (100 per cent.) hydrochloric acid needed to combine with 100 Gm. of calcium carbonate?
2. How many grammes of 31.9 per cent. hydrochloric acid needed to combine with 100 Gm. of calcium carbonate?
3. How many grammes of calcium chloride will be made from 100 Gm. of calcium carbonate?
4. How many grammes of 31.9 per cent. hydrochloric acid needed to combine with 10 Gm. calcium carbonate?

Compare these figures with the recipe given above.

Now let us calculate the manufacture of calcium carbonate. Work out molecular weights of calcium chloride, calcium carbonate, and monohydrated sodium carbonate, and place these molecular weights under the equation (B) given above. Then solve the following problems:

1. How many grammes of calcium carbonate can be made from 111 Gm. of calcium chloride?

2. How many grammes of monohydrated sodium carbonate needed to combine 111 Gm. of calcium chloride?

3. How many grammes of calcium carbonate can be made from 11.1 Gm. of calcium chloride?

4. How many grammes of crystalline sodium carbonate needed to combine 11.1 Gm. of calcium chloride?

Compare these figures with that of the recipe given above.

Remarks on Process.—The solution of the marble and the precipitation of the chalk are explained fully enough in recipe.

Decant supernatant liquid. Supernatant liquid is the clear liquid resting above precipitate. Decant it by gently tilting beaker, being careful to keep precipitate resting quietly at bottom of beaker. Use guiding rod if desired. (See p. 126.)

Wash precipitate to remove all soluble salts, be they either calcium chloride or sodium carbonate. If the former is present, the silver nitrate will give a white, curdy precipitate; if sodium carbonate is there, the calcium chloride will give a white precipitate. If both are washed out and only water passes through the precipitated chalk, it will, of course, produce no precipitate with either reagent.

Dry the precipitate as you did the granulated salts (see p. 1098), only, if desired, more heat can be used than with them—even the heat of a radiator or a drying oven.

MAKE EFFERVESCENT MAGNESIUM CITRATE, U. S. P. 1890

<i>Recipe.</i> —Magnesium carbonate.....	5 Gm.
Citric acid.....	23 Gm.
Sodium bicarbonate.....	17 Gm.
Sugar, in fine powder.....	4 Gm.
Alcohol,	
Distilled water, each, a sufficient quantity.	

Mix the magnesium carbonate intimately with 15 Gm. of citric acid and 2 Cc. of distilled water, so as to form a thick paste. Dry this at a temperature not exceeding 30° C. (86° F.), and reduce it to a fine powder. Then mix it intimately with the sugar, the sodium bicarbonate, and the remainder of the citric acid, previously reduced to a very fine powder. Dampen the powder with a sufficient quantity of alcohol, so as to form a mass, and rub it through a No. 6 tinned-iron sieve. Then dry it, and reduce it to a coarse, granular powder.

Keep the product in well-closed vessels.

Chemical Explanation.—The first reaction is that between magnesium carbonate and citric acid, whereby magnesium citrate ($Mg_3(C_6H_5O_7)_2$) is produced, while carbonic acid gas is expelled.

Owing to the complex formula of magnesium carbonate (which look up on p. 499), the equation representing reaction is omitted, and partially because the amount of citric acid directed is far in excess of the theoretic quantity. In short, the preparation, while made by chemical reaction, is more like a pharmaceutical combination.

The dried acid magnesium citrate is then mixed with sugar, sodium bicarbonate, and more citric acid, great care being taken to prevent chemical action between the last two ingredients. For this reason we moisten the mass before granulation with alcohol, and *not* with

water, since water would dissolve the bicarbonate and the acid, and thus bring about the action which it is desired to retard.

The dry granules, therefore, contain, besides sugar and magnesium citrate, particles of sodium bicarbonate and citric acid resting side by side uncombined. So soon as the dry granules are thrown into water the two chemicals last mentioned combine, with violent effervescence, as shown by the following equation:



Complete this equation. The effervescence is due to the liberation of carbon dioxide.

Remarks on Process.—*Mix the magnesium carbonate and the citric acid by triturating in mortar, having first finely powdered each.*

Magnesium carbonate in blocks can best be powdered by rubbing against a No. 40 or 50 sieve.

No more water than that directed (2 Cc.) need be used to make paste.

Trituration of mass with pestle will hasten the result.

When paste is made, scrape off mass from pestle, using a nickle-plated or horn spatula, if possible. There is great danger of iron contamination if a steel spatula is used.

Dry mass by placing mortar containing it on radiator or at back of a stove.

When dry, powder the magnesium citrate and add the other ingredients.

Moisten with strong alcohol, not using more than 2 Cc. of the alcohol. If the mixture is kneaded sufficiently, a soft mass will result.

Push moist mass through clean coarse sieve, letting granules fall on paper placed beneath.

Dry granules by placing paper on radiator and put into dry bottle without great delay.

MAKE SYRUP OF LIME, U. S. P.

Recipe.—Lime.....	6.5 Gm.
Sugar.....	35.0 Gm.
Water, a sufficient quantity,	
To make..	100 Cc.

Slake the lime by the addition of 4 Cc. of water with the aid of heat, then mix it and the sugar thoroughly in a mortar, so as to form a homogeneous powder; add the mixture to 50 Cc. of boiling water, contained in a bright copper or tinned-iron vessel, and boil for five minutes, constantly stirring. Dilute the liquid with sufficient water to make it measure 95 Cc., and filter through white paper, closely covering the funnel during filtration. Then add through the filter enough water to make the product measure 100 Cc., and mix thoroughly. Keep syrup in well-stoppered bottles.

Chemical Explanation.—Lime is only sparingly soluble in water, lime water, the saturated solution, containing only one-sixth of 1 per cent. of lime.

On the other hand, lime is quite soluble in water if first combined with sugar; for, in this case, a chemical combination takes place be-

tween the lime and the sugar. Such combination is called calcium saccharate, although this is scarcely correct, inasmuch as sugar contains no acid, but is an aldehyde or ketone alcohol, and the calcium compound is, therefore, similar to sodium alcoholate (see p. 655) and sodium carbolate (see p. 758).

Since the chemistry of this calcium saccharate is rather vague, no attempt will be made to work out the equation of manufacture.

Remarks on Process.—*The bright copper vessel* can be a well-scoured copper water-bath. Porcelain dish not advisable, since the lime will attack the glazing.

Filter diluted liquid through white paper. The filtrate should be perfectly transparent, and must be returned through filter-paper until it is clear.

Strain through piece of muslin previously well wetted with water.

In evaporating filtrate do not let it get too concentrated, for then there results a sticky mass, which is difficult to remove.

In boiling the solids with water, be sure to add extra water to make up for loss of water through evaporation. If this is not heated, a sticky and difficultly soluble mass results.

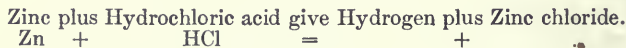
MAKE SOLUTION OF ZINC CHLORIDE, U. S. P.

<i>Recipe.</i> —Zinc, granulated.....	12.0 Gm.
Hydrochloric acid.....	42.0 Gm.
Nitric acid.....	0.6 Gm.
Precipitated zinc carbonate.....	0.6 Gm.
Distilled water, a sufficient quantity,	

To make about. . 50 Gm.

To the zinc, contained in a glass or porcelain vessel, add 8 Cc. of distilled water; then gradually add the hydrochloric acid, and digest until reaction ceases and the acid is saturated. Pour off the solution, add the nitric acid, and heat the solution at a temperature not exceeding 115° C. (239° F.), until a portion, if removed and cooled, solidifies. Allow it to cool, and dissolve the solidified mass in a sufficient quantity of distilled water to make the product weigh 50 Gm. Then add the precipitated zinc carbonate, agitate the mixture occasionally during twenty-four hours, and set it aside until it has become clear by subsidence. Finally, separate the clear solution by decantation or by means of a siphon.

Chemical Explanation.—Zinc chloride is made by treating zinc with hydrochloric acid, the reaction being as follows:



Complete this equation.

As this solution is supposed to be made from commercial zinc, and as this *usually* contains iron, the iron chloride produced when such metal is dissolved in hydrochloric acid must be removed from the solution. This is accomplished by adding nitric acid, evaporating the solution, and finally fusing the dry mass. Neither nitric acid nor fusion affects the zinc chloride, while nitric acid and fusion convert the ferric chloride into insoluble ferric oxide, and when the fused mass is treated with

water, the zinc chloride dissolves, leaving the ferric oxide as an insoluble residue.

The solution is too caustic to be filtered through paper, so the ferric oxide is removed by sedimentation, precipitated zinc carbonate being added to aid in the clarification.

Calculations.—Figure out the molecular weights of hydrochloric acid and of zinc chloride, and give the atomic weight of zinc, and with these figures work out the following examples:

1. How many grammes of zinc and absolute (100 per cent.) hydrochloric acid needed to make 136 Gm. zinc chloride?

2. How many grammes of 31.9 per cent. hydrochloric acid needed to make 136 Gm. zinc chloride?

3. How much zinc chloride in 50 Gm. solution of zinc chloride, U. S. P., if this solution contains 50 per cent. of the salt?

4. How much zinc and 31.9 per cent. hydrochloric acid needed to make 25 Gm. of zinc chloride?

5. Then how much zinc and 31.9 per cent. hydrochloric acid needed to make 50 Gm. solution of zinc chloride, U. S. P.?

Compare with quantities given in above recipe.

Remarks on Process.—Place zinc in Erlenmeyer flask, and add thereto the water and the acid.

Digest mixture by letting stand several hours in a warm place—say, on a radiator or at back of stove. Note evolution of hydrogen gas as the zinc dissolves in the hydrochloric acid.

Pour off solution (instead of filtering) into a beaker or 8-ounce wide-mouthed bottle, and evaporate to dryness in small evaporating dish on sand-bath. If the dish is not large enough to hold all the solution at one time, add part at first, and when this has evaporated, add the rest. When mass is dry,

Fuse cautiously, so as to not exceed the pharmacopœial temperature, else the zinc chloride loses chlorine and decomposes. A thermometer is not necessary, however, if the mass is cautiously fused over a Bunsen burner

Dissolve fused mass in about 25 Cc. distilled water, and transfer to tared Erlenmeyer flask. Wash out dish with 5 to 10 Cc. water, and use this water for bringing the weight of the solution in Erlenmeyer up to 50 Gm.

Separate clear solution by decantation. A syphon is suitable only when a large quantity is made.

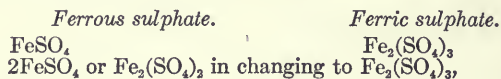
MAKE SOLUTION OF TERSULPHATE OF IRON, U. S. P.

<i>Recipe.</i> —Ferrous sulphate, in clear crystals.....	62.5 Gm.
Sulphuric acid.....	12.0 Gm.
Nitric acid,	
Distilled water, each, a sufficient quantity,	
To make. . .	125 Gm.

Add the sulphuric acid to about 30 Cc. of distilled water in a capacious porcelain dish, heat the mixture to nearly 100° C. (212° F.), then add 7 Gm. of nitric acid, and mix well. Divide the ferrous sulphate, coarsely powdered, into four equal portions, and add these portions, one at a time, to the hot liquid, stirring after each addition until effervescence ceases. When all the ferrous sulphate is dissolved,

if the solution has acquired a black color, add nitric acid, a few drops at a time, heating and stirring until it no longer causes red fumes to be evolved, and the solution assumes a clear, reddish-brown color; then boil the liquid until it is free from nitric acid. Lastly, add enough distilled water to make the product weigh 125 Gm. Filter if necessary.

Chemical Explanation.—This solution is prepared by the oxidation of green ferrous sulphate, FeSO_4 , to red-brown ferric sulphate, $\text{Fe}_2(\text{SO}_4)_3$, through oxidizing action of nitric acid in the presence of sulphuric acid. The need of this extra sulphuric acid is shown in comparison of the formulas of ferrous sulphate and ferric sulphate respectively:

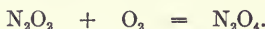


requiring an extra SO_4 group.

The oxidizing action of nitric acid is fully explained on p. 420, and there it is shown that every two molecules of nitric acid give three atoms of oxygen in oxidations. With these hints construct the equation representing the reaction, viz.:

Ferrous sulphate plus sulphuric acid plus nitric acid equals ferric sulphate plus nitrogen dioxide plus water.

Note that the red fumes given off during the reaction are not N_2O_2 , but are N_2O_4 , which is formed the moment the colorless gas N_2O_2 , comes in contact with air, viz.:



Calculations.—Figure out molecular weight of crystallized ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), sulphuric acid, nitric acid, and ferric sulphate, and with these figures work out the following examples:

1. How many grammes crystalline ferrous sulphate, 100 per cent. sulphuric acid, and 100 per cent. nitric acid needed to make 1200 Gm. of ferric sulphate?

2. How many grammes 91 per cent. sulphuric acid needed to make 1200 Gm. of ferric sulphate?

3. How many grammes 68 per cent. nitric acid needed to make 1200 Gm. of ferric sulphate?

4. How many grammes of ferric sulphate are contained in 125 Gm. of solution of ferric sulphate, U. S. P. (36 per cent.).

5. How many grammes of crystalline ferrous sulphate, 91 per cent. sulphuric acid, and 68 per cent. nitric acid needed to make 45 Gm. ferric sulphate?

6. Then how many grammes of these three constituents needed to make 125 Gm. of solution of ferric sulphate, U. S. P.?

Compare with quantities in official recipe.

Remarks on Process.—*Mix sulphuric acid and water in tared pint evaporating dish, carefully pouring acid into water and not the reverse.*

Add the ferrous sulphate gradually, letting reaction end before adding another of the four portions.

Add the nitric acid fairly generously. If you get an excess, it can be driven off by heating mixture. Do not add too much, however, as it will be wasteful of the acid, and will also require long heating to drive it all off.

If the liquid is black, it means an insufficiency of nitric acid. Add some, and the red tint will come, accompanied by evolution of N_2O_4 fumes.

Boil until free from nitrous odor, that is, the choking odor of nitric acid and of N_2O_4 . Experience will readily teach you the odor.

Add water to make product weigh 125 Gm. To do this easily, be sure to tare the dish before starting.

MAKE SOLUTION OF FERRIC CITRATE, U. S. P.

Recipe.—Solution of ferric sulphate.....	52.5 Gm.
Citric acid.....	15.0 Gm.
Ammonia water.....	44.0 Cc.
Water, a sufficient quantity,	

To make. . 50 Gm.

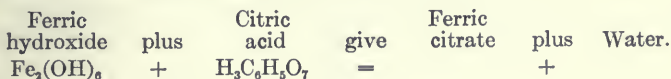
Mix the ammonia water with 150 Cc. of cold water, and the solution of ferric sulphate with 500 Cc. of cold water. Add the latter solution slowly to the diluted ammonia water, with constant stirring. Pour the mixture on a wet muslin strainer, and allow the liquid to run off and the precipitate to drain. Then remove the moist mass from the strainer, mix it well with 300 Cc. of cold water, again pour it on the strainer, and let it drain. Repeat this washing with several successive portions of cold water in the same manner, until the washings cease to produce more than a slight cloudiness with barium chloride test-solution. Then allow the precipitate to drain completely, transfer it to a porcelain capsule, add the citric acid, and heat the mixture, on a water-bath, to 60°C . (140°F .), stirring constantly until the precipitate is dissolved. Lastly, filter the liquid, and evaporate it, at the above-mentioned temperature, until it weighs 50 Gm.

Chemical Explanation.—The first stage of the process of the precipitation of ferric hydroxide from the solution of ferric sulphate is by the following reaction:



Complete this equation.

The ferric hydroxide is freed from ammonium sulphate by washing with water, and is then treated with citric acid, which converts the moist ferric hydroxide into solution of ferric citrate, viz.:



Complete this equation.

Calculations.—As the ferric citrate is a rather variable product, containing excess of citric acid, attempts to figure out proportions by molecular weights are of no avail.

Remarks on Process.—Place the diluted ammonia in a quart fruit-jar, and pour therein the solution of ferric sulphate diluted with 500 Cc. of water directed by U. S. P. Note the red-brown ferric hydroxide precipitates as a magma (p. 141). Note that this is the antidote for arsenic (p. 564).

Pour mixture on wet muslin strainer, spread upon a straining rack. Unless the strainer is of fine mesh, the precipitate will run through. If too fine, the draining will be a slow operation. In large operations it is well to have three strainers on racks placed one above the other, and the magma is poured into the upper one. Most of the precipitate which passes through the upper strainer is caught by the middle one, and whatever does get through the middle one is caught by the lowest one. In laboratory work the laying of a 6-inch filter-paper flat on the strainer holds the magma excellently. Collect drippings in pint evaporating dish and return to strainer whatever passes through containing magma. The drippings should be clear and colorless.

Remove mass from strainer while washing. This part of the pharmacopœial process is a smeary process in the laboratory operation. In large operations, where the strainer is fine duck attached to the frame, the magma can be easily scraped from the strainer with a big spoon; but in the laboratory process it is usually better to wash magma while on the strainer, and that either with a syphon (p. 126) or by continuous washing with inverted flask (p. 124). In washing thus, be careful not to disturb the magma so that it will run through strainer.

Wash until drippings give no precipitate with barium chloride. That is, until free from ammonium sulphate.

Transfer magma to porcelain capsule—that is, to evaporating dish, by scraping off the strainer with spatula or with a spoon.

Do not heat magma and citric acid above 60° C.—that is, even the full heat of a water-bath is apt to decompose the citrate.

Filter solution and evaporate in tared pint evaporating dish.

MAKE FERRIC CITRATE, U. S. P. 1890 (IN SCALES)

Recipe.—Solution of ferric citrate..... 25 Gm.

Evaporate the solution on a water-bath, at a temperature not exceeding 60° C. (140° F.), to the consistence of syrup, and spread it on plates of glass, so that, when dry, the salt may be obtained in scales.

Keep the product in well-stoppered bottles, protected from light.

Remarks on Process.—Solution of ferric citrate (U. S. P. 1890) is about 50 per cent. solid ferric citrate, and the 25 Gm. should, therefore, yield about 12½ Gm. of the scaled salt.

Evaporate at low temperature (60° C.) to avoid decomposition.

Spread syrupy liquid on sheet of glass or on a pill-tile. Be sure to spread thinly, using glass rod in getting layer smooth. If poured on thickly, opaque scales result.

Leave liquid on plates until perfectly dry, using no heat whatever. Artificial heat—even a radiator—causes blisters.

If properly prepared, the dry salt will scale off the pill-tile without scraping, and that in the form of beautiful shining garnet scales.

MAKE MASS OF FERROUS CARBONATE

Recipe.—Ferrous sulphate, in clear crystals..... 20.0 Gm.
 Monohydrated sodium carbonate..... 9.2 Gm.
 Clarified honey..... 7.6 Gm.
 Sugar, in coarse powder..... 5.0 Gm.
 Syrup,
 Distilled water, each, a sufficient quantity,
 To make.. 20 Gm.

Dissolve the ferrous sulphate and the monohydrated sodium carbonate, each separately, in 40 Cc. of boiling distilled water, and, having added 4 Cc. of syrup to the solution of the iron salt, filter both solutions, and allow them to become cold. Introduce the solution of monohydrated sodium carbonate into a bottle having a capacity of about 100 Cc., and gradually add the solution of the iron salt, rotating the bottle constantly or frequently, until carbonic acid gas no longer escapes. Add a sufficient quantity of distilled water to fill the bottle; then cork it and set it aside, so that the ferrous carbonate may subside. Pour off the supernatant liquid, and, having mixed syrup and distilled water in the proportion of one volume of syrup to nineteen volumes of distilled water, wash the precipitate with the mixture by decantation until the washings no longer have a saline taste. Drain the precipitate on a muslin strainer, and express as much of the water as possible. Lastly, mix the precipitate at once with the honey and sugar, and, by means of a water-bath, evaporate the mixture in a tared dish, with constant stirring, until it is reduced to 20 Gm.

Chemical Explanation.—When solutions of ferrous sulphate and sodium carbonate are mixed, ferrous carbonate precipitates in the form of a slate-gray solid, viz.:



Complete this equation.

Ferrous carbonate is a very unstable substance, being converted by action of the atmosphere into red-brown subcarbonate of iron—a mixture of ferrous carbonate and ferric oxide. This change will be noted during the manufacture of the mass in all portions not protected from atmospheric action.

The presence of saccharine matter retards this change, and this explains why the precipitate is washed with water containing syrup, and why the finished product is preserved with sugar and honey. The precipitation is directed to be performed in a flask which is to be kept closed as much as possible, in order to lessen atmospheric action. It will be seen above that the other product of the reaction is sodium sulphate, and the precipitate is to be washed free from this with water and syrup.

Calculations.—The pharmacopœia directs no definite percentage of ferrous carbonate in the mass, since the handling of the precipitate gives much chance for loss and uncertainty.

We will, however, calculate the amount of ferrous carbonate the mass should contain.

Estimate molecular weight of crystalline ferrous sulphate ($\text{FeSO}_4\cdot 7\text{H}_2\text{O}$), of monohydrated sodium carbonate ($\text{Na}_2\text{CO}_3\cdot \text{H}_2\text{O}$), and of ferrous carbonate, and from the figures work out the following examples:

1. How many grammes monohydrated sodium carbonate will combine with 278 Gm. crystalline ferrous sulphate?

2. How many grammes ferrous carbonate can be made from 278 Gm. crystalline ferrous sulphate?

3. How many grammes of monohydrated sodium carbonate will combine with 20 Gm. of crystalline ferrous sulphate?

4. How many grammes ferrous carbonate can be made from 20 Gm. crystalline ferrous sulphate?

5. How many grammes of ferrous carbonate should there be in 20 Gm. mass of ferrous carbonate?

6. Then what percentage of ferrous carbonate should there be in mass of ferrous carbonate?

Remarks on Process.—*Dissolve the ferrous sulphate* in water in one beaker.

Dissolve the sodium carbonate in water in the other beaker.

Filter the sodium carbonate solution into Erlenmeyer flask, to which a cork should have been previously fitted.

Filter the ferrous sulphate solution into the beaker just emptied after it has been washed and dried.

Cork the Erlenmeyer flask as soon as convenient, after adding the ferrous sulphate solution, but be careful not to allow enough carbon dioxide to collect to explode the flask. Open flask from time to time to let collected gas escape, but only for a moment each time. Be sure to fill flask with water, as directed by U. S. P.

Wash precipitate on same kind of strainer used for ferric hydroxide in making ferric citrate.

Evaporate mixture in tared capsule, that is, in an evaporating dish, the weight of which should first be ascertained.

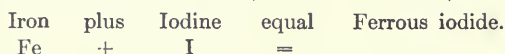
MAKE SYRUP OF FERROUS IODIDE, U. S. P.

<i>Recipe.</i> —Iron, in the form of fine, bright wire, and cut into small pieces.....		1.25 Gm.
Iodine.....		4.15 Gm.
Diluted hypophosphorous acid.....		2.00 Cc.
Sugar.....		60.00 Gm.
Distilled water, a sufficient quantity;		

To make.. 100 Gm.

Introduce the iron into a flask of thin glass, having a capacity of about 50 Cc., add to it 15 Cc. of distilled water, and afterward the iodine. Shake the mixture occasionally, checking the reaction, if necessary, by the affusion of cold water, and, when the solution has acquired a greenish color and has lost the odor of iodine, heat it to boiling and add at once 5 Gm. of the sugar; when this has dissolved, filter the solution into the remainder of the sugar contained in a porcelain dish. Rinse the flask and iron wire with $12\frac{1}{2}$ Cc. of distilled water and pass the washings through the filter into the sugar. Stir the mixture with a porcelain or wooden spatula, heating the liquid on a water-bath until complete solution is effected, and, having passed the syrup through a clean muslin strainer into a tared bottle, add the diluted hypophosphorous acid and sufficient distilled water to make the product weigh 100 Gm.

Chemical Explanation.—Ferrous iodide is made by the direct combination of the two elements, iron and iodine, viz.:



Complete this equation.

Calculations.—Figure out the atomic and molecular weights involved in the above equation, and then solve the following problems:

1. How many grammes iron needed to make 310 Gm. ferrous iodide?
2. How many grammes iron needed to make 5 Gm. ferrous iodide?
3. How many grammes iodine needed to make 310 Gm. ferrous iodide?
4. How many grammes iodine needed to make 5 Gm. ferrous iodide?
5. How many grammes ferrous iodide in 100 Gm. syrup ferrous iodide (5 per cent.)?
6. Then how many grammes of iron and iodine needed to make 100 Gm. syrup of ferrous iodide?
7. Compare these figures with amounts given in official recipe. The slight excess of iron is used to insure complete combination of the iodine.

Remarks on Process.—*The iron used had best be that form known in commerce as "card teeth."*

Place iron either in Erlenmeyer flask or in large test-tube.

After adding the iodine it is well to plug flask with a pledget of cotton to prevent loss of iodine by volatilization.

If reaction is slow on starting, very gentle heat may be employed. Very gentle is meant, as anything more will cause loss by volatilization.

If reaction becomes violent, check it by holding flask under running water.

Have the sugar in tared pint evaporating dish, as finished product must be brought up to a certain weight.

Keep in completely filled bottles, else it turns brown with decomposition of the ferrous iodide. One-ounce bottles well corked are indicated.

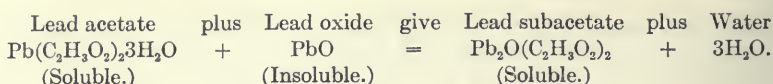
MAKE SOLUTION OF LEAD SUBACETATE, U. S. P.

<i>Recipe.</i> —Lead acetate.....	18 Gm.
Lead oxide.....	11 Gm.
Distilled water, a sufficient quantity,	
To make..	<u>100 Gm.</u>

To the finely powdered lead oxide contained in a porcelain dish, of about half-liter capacity, add slowly and in portions, with constant stirring, the lead acetate which has been previously dissolved in 70 Cc. of boiling distilled water, and boil the liquid for half an hour, with occasional stirring. Finally, when cool, filter the solution, and add sufficient distilled water, which has been previously boiled and cooled, to make the finished product weigh 100 Gm. Keep the solution in well-stoppered bottles.

Chemical Explanation.—As explained on p. 561, a "sub" salt is a basic salt—that is, a combination of a salt of a metal with the oxide of the same metal.

In the preparation under consideration the "sub" salt is made by direct combination of the salt and the oxide, lead acetate and lead oxide being boiled together with the formation of lead subacetate, as shown in the following equation:



Calculations.—Figure out molecular weights of the three lead preparations given above, and from these figures work out the following examples:

1. How many grammes lead acetate needed to make 548 Gm. lead subacetate?

2. How many grammes lead acetate needed to make 25 Gm. lead subacetate?

3. How many grammes lead oxide needed to make 548 Gm. lead subacetate?

4. How many grammes lead oxide needed to make 25 Gm. lead subacetate?

5. How many grammes lead subacetate in 100 Gm. solution of lead subacetate (25 per cent.)?

6. Then how many grammes of lead subacetate and lead oxide needed to make 100 Gm. solution of lead subacetate?

Compare with amounts directed in official recipe.

Remarks on Process.—*Dissolve lead acetate* in pint flask.

Pass the lead oxide through a No. 80 sieve before weighing, and then put into tared pint evaporating dish.

Boil mixture a half-hour, during which time a large amount of the insoluble lead oxide passes into the soluble lead subacetate.

All the lead oxide will not dissolve, however, and that is the reason an excess over the theoretic amount is directed in official recipe.

Add hot water to make up the loss of evaporation, otherwise the mixture will evaporate to dryness during the half-hour's boiling.

Make finished product weigh 100 Gm. by adding enough *freshly boiled* water. Even distilled water contains carbon dioxide if allowed to stand, and this carbon dioxide will precipitate the lead as lead carbonate; so boil the water to drive off the dissolved carbon dioxide before using it as a diluent.

Filter in closely covered funnel and keep in well-stoppered bottles to prevent atmospheric action. The air contains carbon dioxide, and this precipitates the lead, as mentioned above.

MAKE LEAD PLASTER, U. S. P. 1890

Recipe.—Lead oxide..... 16 Gm.
 Olive oil..... 30 Gm.
 Water, a sufficient quantity.

Mix the lead oxide, previously passed through a No. 80 sieve, intimately with about one-half of the olive oil, by trituration, and add the mixture to the remainder of the oil contained in a bright copper boiler of a capacity equal to at least four times the bulk of the ingredients. Then add 5 Cc. of boiling water, and boil the whole together, over a fire, constantly stirring with a wooden spatula, until a small portion, when dropped into cold water, is found to be pliable and tenacious. From time to time add a little water to replace that lost by evaporation. When the contents of the boiler have acquired

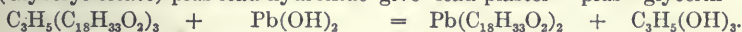
a whitish color and are perfectly homogeneous, transfer them to a vessel containing warm water, and as soon as the mass has sufficiently cooled, knead it well with the water so as to remove the glycerin, renewing the water from time to time, as long as it may be necessary. Finally, divide the mass into rolls of suitable size.

Chemical Explanation.—As learned on p. 737, when an oil—which is the combination of glycerin with oleic acid and other acids—is treated with the hydroxide of a metal, glycerin is set free and a metallic oleate is formed. Thus, if an oil is treated with soda (NaOH), sodium oleate or hard soap is formed; if the same oil is treated with potassa (KOH), potassium oleate or soft soap is obtained.

Therefore, when olive oil is boiled with lead oxide in the presence of water (thus forming lead hydroxide, viz., $\text{PbO} + \text{H}_2\text{O} = \text{Pb}(\text{OH})_2$), lead oleate is produced by the following equation:

Olive oil
(Olein)

(Glyceryl oleate) plus lead hydroxide give lead plaster plus glycerin



Complete this equation.

The result of the reaction is a viscid mass consisting chiefly of lead oleate, and called lead plaster, and by kneading this lead plaster with water the glycerin is removed.

As the olive oil contains traces of glyceryl palmitate and glyceryl stearate, the finished plaster contains some lead palmitate and lead stearate; hence it is not a definite chemical.

Calculations.—Since we are not dealing with definite chemicals, the molecular proportions of this reaction cannot be easily worked out; hence they are omitted.

Remarks on Process.—*Triturate the sifted lead oxide* in mortar with about half the oil, rubbing it with small portions of the oil at a time, thus using the oil to wash the oxide into the rest of the oil in the evaporating dish. Be sure to get all the oxide out of the mortar into the dish.

The plaster should be cooked in a copper boiler, since the lead is apt to attack glass or porcelain, dissolving off some. For the small amount we are making, however, the pint evaporating dish can be used.

Boil mixture in dish over Bunsen burner flame on wire gauze, stirring with wooden paddle. Both glass rod and steel spatula are tabooed—the first for fear the lead might attack it; the steel spatula lest the oleic acid attack it.

Replace water lost by evaporation, and that only cautiously, adding boiling hot water. The temperature of the boiling plaster mass is considerably above 100°C ., and if cold water is added, steam will be developed with explosive suddenness, sputtering the hot plaster mass in all directions, and rendering the operator liable to severe burns.

The mass is finished when a drop solidifies in water.

After kneading out the glycerin from the finished matter the glycerin may be recovered from the watery solution—from the wash-water—by evaporating off the water at very low heat.

Use hydrant water for the kneading.

MAKE RESIN PLASTER, U. S. P. 1890

<i>Recipe.</i> —Resin, in fine powder.....	3.5 Gm.
Lead plaster.....	20.0 Gm.
Yellow wax.....	1.5 Gm.

Melt the lead plaster and yellow wax together with a gentle heat; then add the resin, and, when it is melted, mix the mass thoroughly.

As this mixture is a galenic preparation, there will be no molecular calculations.

Remarks on Process.—Melt the mixture in pint evaporating dish and strain into a tin or tinned-iron dipper.

SPREAD A RESIN PLASTER 4 × 6

The spreading of plasters has become a lost art, but every pharmacist should have sufficient practice to know how to spread one in an emergency.

A plaster is usually spread either on leather (the commercial variety called split skin) or on muslin. This, for convenience, we will call the foundation.

For laboratory practice a sheet of paper can be used. Let the material on which the plaster is spread be sufficiently large to permit a margin of at least an inch all around after the plaster is spread.

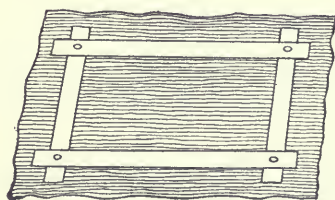


Fig. 245.

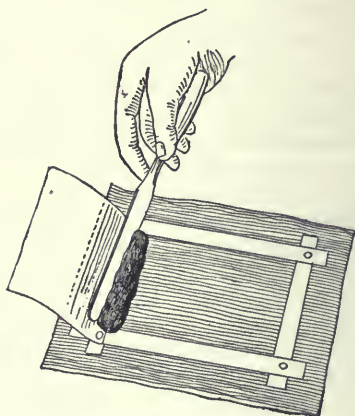


Fig. 246.

Figs. 245 and 246.—Spreading a plaster.

The leather, cloth, or paper is carefully stretched over a perfectly level board (the clean counter will do, if it can be pierced with pins), and fastened down at margins with the brass or nickle-headed tacks known commercially as thumb tacks. With paper, this stretching is unnecessary.

Strips of fairly heavy linen paper are cut with an exact, sharp margin, the strips being about two inches wide and eight inches long. The strips are placed on the stretched plaster paper in such a way as to form an exactly even frame inclosing a space the size of the plaster directed to be spread; in this case, four by six inches. The strips are fastened at each corner by thumb tacks, and when made secure

the tacks originally used for stretching the leather—if leather is used—may be removed.

This done, the plaster mass in tin dipper is cautiously fused, and when melted, small portions of the molten mass are taken out with a spatula and smeared between strips and foundation in very thin layer, in order to fasten the strips to the foundation. Then the spatula is carefully cleaned by passing through a flame of Bunsen burner and wiping off on paper. The foundation should be placed with the length running with the length of the counter, and on this, at narrow end to the left, should be poured the fused mass, so as to cover about one-third the length of the plaster. Then, without loss of time, the spatula should be warmed by passing through the flame, and being held in the right hand with blade slightly inclined forward, but perfectly level, about one-quarter to one-half inch above the whole width of plaster, the spatula should be moved rapidly and firmly to the right, pushing before it the still molten plaster mass and leaving behind a smooth, shining spread plaster.

Remarks on Process.—The *spatula* should be at least eight inches long, and should not be sufficiently heated to decompose the plaster mass.

The *plaster mass* should be cautiously fused—not too hot to decompose or to run through the foundation, and yet limpid enough to remain fused until the plaster is entirely spread.

Running the spatula over the fused mass is the delicate part of the process. It must be done as quickly as possible lest the mass solidifies before the process is complete. The spatula must be held perfectly steady else the plaster will be uneven.

In fastening the foundation to the counter place it an inch or so from the edge of the counter, thus leaving a rest for the hand holding the spatula as it passes over the mass.

Remove the strips from the plaster by taking out thumb tacks and pulling off. If the mass is brittle, passing a warm spatula over the strips sometimes aids in loosening them, though this must be done with caution, lest the plaster itself become smeary. The strips removed, a clearly defined plaster should remain, the excess of the mass having been removed with the strips.

The *excess of mass on strips* can be scraped off the strips with warm spatula and left for use another time.

There is little use to attempt to redeem a poorly spread plaster by “reironing”; that is, by trying to smooth it with a warm spatula. The best thing to do is to scrape the mass from the foundation with warm spatula and try over again.

Other plans for plaster spreading by hand have been followed with more or less success. Thus, for the paper strips, strips of pasteboard one-quarter inch thick have been used, and the entire space within the frame filled with the molten mass, the excess being scraped off with the warm spatula. Others cut the frame from an entire sheet of paper.

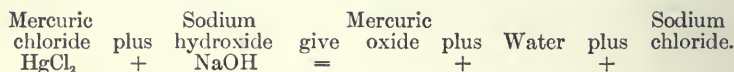
For *machine spreading of plasters*, see p. 353.

MAKE YELLOW MERCURIC OXIDE

Recipe.—Corrosive mercuric chloride..... 10 Gm.
Sodium hydroxide..... 4 Gm.
Distilled water, a sufficient quantity.

Dissolve the corrosive mercuric chloride in 100 Cc. of warm distilled water, and filter the solution. Dissolve the sodium hydroxide (which should contain at least 90 per cent. of pure anhydrous sodium hydroxide) in 100 Cc. of cold distilled water, and into this solution pour gradually, and with constant stirring, the solution of corrosive mercuric chloride. Allow the mixture to stand for an hour at a temperature of about 30° C. (86° F.), stirring frequently. Then decant the supernatant, clear liquid from the precipitate, and wash the latter repeatedly by the addition and decantation of portions of distilled water, using 100 Cc. of water each time. Collect the precipitate on a strainer, and continue the washing with warm distilled water until a small portion of the washings, when poured on a little mercuric chloride test-solution, no longer produces a yellowish turbidity at the line of contact of the two liquids. Then allow the precipitate to drain, and dry it between sheets of bibulous paper, in a dark place, at a temperature not exceeding 30° C. (88° F.), and keep it in well-stoppered bottles, protected from light.

Chemical Explanation.—When solutions of mercuric chloride and of sodium hydroxide are combined, yellow mercury oxide is precipitated by the following reaction:



Complete this equation.

Calculations.—Work out molecular weights of mercuric chloride, sodium hydroxide, and mercuric oxide (HgO). From these figures work out the following examples:

1. How many grammes of mercuric oxide can be made from 271 Gm. mercuric chloride?
2. How many grammes of mercuric oxide can be made from 10 Gm. mercuric chloride?
3. How many grammes of sodium hydroxide (100 per cent.) will combine with 271 Gm. mercuric chloride?
4. How many grammes of sodium hydroxide (85 per cent.) will combine with 271 Gm. mercuric chloride?
5. How many grammes of sodium hydroxide (85 per cent.) will combine with 10 Gm. mercuric chloride?

Compare with figures given above, and weigh your finished mercuric oxide to see if it agrees with the theoretic yield.

The excess of soda directed in U. S. P. is to insure complete precipitation of the mercury, the surplus soda being washed out of the precipitate with water.

Remarks on Process.—*Dissolve the mercuric chloride* in Erlenmeyer flask and filter into an 8-ounce wide-mouthed bottle.

Dissolve the soda in pint flask, and into this pour the mercuric chloride solution.

Wash the precipitate by decantation as far as possible, and finally throw precipitate on plain filter, instead of on strainer, as the U. S. P. directs. Were we making the pharmacopœial quantity (ten times our present amount), a strainer would be indicated, but with the small amount we have, a plain filter will suffice.

Wash until washings no longer respond with mercuric chloride test-solution—that is, until free from soda. If soda is present in water, a yellow precipitate (HgO) is produced with mercuric chloride solution.

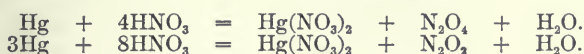
In testing for soda place about one inch of the mercuric chloride solution in test-tube and pour thereon very gently, along tilted test-tube, the washings, so as to permit the washings to float on surface of the mercuric chloride solution, thus forming two layers; where the two layers join—"the point of contact"—the yellow color appears.

MAKE OINTMENT OF MERCURIC NITRATE, U. S. P.

<i>Recipe</i> .—Mercury.....	7.0 Gm.
Nitric acid.....	17.5 Gm.
Lard, free from water.....	76.0 Gm.
	To make about.. 100 Gm.

Heat the lard in a capacious glass or porcelain vessel to a temperature of 105° C. (221° F.), then withdraw the heat, and gradually add 7 Gm. of the nitric acid. When the reaction moderates, reapply the heat until effervescence ceases, and allow the mixture to cool to about 40° C. (104° F.). Having dissolved the mercury in the remainder of the nitric acid, using sufficient heat to prevent the solution from crystallizing, add this solution to the lard mixture. When the mass begins to congeal, stir it thoroughly with a wooden spatula until it is of a bright citrine color. Contact with metallic utensils should be avoided.

Chemical Explanation.—In this preparation two distinct reactions occur: In the first place, mercury dissolves in nitric acid to make mercuric nitrate by either of the following equations:



Complete these equations.

Then the white lard is converted into the lemon yellow solid elaidin when the nitric acid is added to it.

Elaidin has the same empiric formula as has the olein of the oil, and since the chemistry of the change is complicated (see p. 727), no molecular weight estimation will be made.

Calculations.—As just mentioned, no calculation can be made on the conversion of olein of the lard into elaidin, nor is the amount of nitric acid used in dissolving the mercury in accord with molecular proportions; hence the amount will not be estimated.

Remarks on Process.—*Put lard in pint evaporating dish* and heat to 105° C. on wire gauze.

A water-bath will not do, as the oil must be heated to 105° C. in order to get reaction started.

Add the nitric acid gradually, not allowing heat to get too intense. If reaction has gone nicely, an orange-colored liquid results; if overheated, the lard turns chocolate brown.

Reapply heat until effervescence ceases. This must be done cautiously, else the mass will turn dark. It must be heated enough, or else the finished product will "work," frothing out of container.

Dissolve the mercury in nitric acid in Erlenmeyer flask. Better start it before treating the lard with nitric acid.

If mercuric nitrate solution crystallizes, warm it before mixing with the elaidin mixture.

Use glass rod during the elaidin reaction, and employ a wooden paddle for mixing in the mercuric nitrate solution.

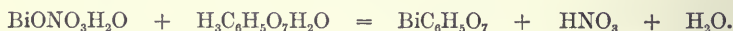
MAKE BISMUTH CITRATE

<i>Recipe.</i> —Bismuth subnitrate.....	10.0 Gm.
Citric acid.....	7.5 Gm.
Distilled water, a sufficient quantity.	

Mix the bismuth subnitrate and the citric acid with 40 Cc. of distilled water, and heat on a bath of boiling water, with frequent stirring, until a drop of the mixture yields a clear solution with ammonia water. Then add 500 Cc. of distilled water, allow the suspended matter to deposit, wash the precipitate, first by decantation, and afterward on a strainer, with distilled water, until the washings are tasteless, and dry the residue at a gentle heat.

Chemical Explanation.—In this preparation the bismuth subnitrate is converted into bismuth citrate by boiling with citric acid, and affords an interesting illustration of the futility of the old terms, “strong acids” and “weak acids.” According to that doctrine, nitric acid is a much “stronger” acid than citric acid, and yet in this case merely boiling of the subnitrate with citric acid causes the driving out of the nitric acid. This is made possible by the fact that nitric acid is a volatile substance, while citric acid is fixed; hence the boiling gives the nitric acid the opportunity for volatilizing, and the citric acid at the same time combines with the metal.

The equation of the change is as follows:



Complete this equation.

Calculations.—1. How much citric acid will combine with 305 Gm. bismuth subnitrate?

2. How much citric acid will combine with 10 Gm. bismuth subnitrate?

3. How much bismuth citrate can be made from 305 Gm. bismuth subnitrate?

4. How much bismuth citrate can be made from 10 Gm. bismuth subnitrate?

Remarks on Process.—*Boil the subnitrate* and citric acid with water in pint flask.

Boil until drop of mixture gives clear solution with ammonia. Both the subnitrate and the citrate are insoluble in water, but, while the subnitrate will not dissolve in ammonia water, the citrate does. Hence, when all the subnitrate is converted into citrate, the entire insoluble precipitate will dissolve in ammonia water, and as long as a part of the precipitate does not dissolve, it means that there is still some unchanged bismuth subnitrate and the boiling must be continued.

Carry out this ammonia solubility test by dipping glass rod into the mixture in flask, and then carefully immersing same into ammonia water in a test-tube. The milky drops on the rod will be dissolved so soon as it is placed in ammonia if the conversion is complete. A faint turbidity, however, is permissible, since it seems practically impossible to convert the last traces of the subnitrate into the soluble citrate.

Add 500 Cc. water to wash out last traces of nitric and citric acid. In this case, since the pint flask holds but 450 Cc., filling the flask with water will be sufficient.

Wash precipitate by decantation, then on strainer, that is, just as you did ferric hydroxide. With the small amount made with the above quantities, collecting and washing on plain filter (6-inch paper) will suffice.

Dry residue at gentle heat—say, on top of radiator.

MAKE BISMUTH AND AMMONIUM CITRATE

Recipe.—Bismuth citrate..... 10 Gm.
Ammonia water,
Distilled water, each, a sufficient quantity.

Mix the bismuth citrate with 20 Cc. of distilled water and rub it to a smooth paste, heat the mixture on a water-bath, and gradually add ammonia water until the salt is dissolved and the liquid is neutral or has only a faintly alkaline reaction. Then filter the solution, evaporate it on a water-bath to a syrupy consistence, and spread it upon plates of glass, so that, when dry, the salt may be obtained in scales. Keep the product in amber-colored, well-stoppered bottles, protected from light.

Chemical Explanation.—As mentioned in preceding process, bismuth citrate is insoluble in water, but it does dissolve in ammonia water, forming with it a loose chemical combination, which, when scaled, is soluble in water.

Calculations.—As the combination between the bismuth citrate and the ammonia is very indefinite, no attempt will be made to work out molecular proportions.

Remarks on Process.—Rub bismuth citrate to smooth paste, by placing in pint evaporating dish and triturating with water, gradually added, with the pestle.

Gradually add ammonia water until the salt is dissolved—10 Cc. usually suffices. An excess works no harm, since it dissipates during the evaporation. While not specified by the pharmacopœia, the addition of a few drops of ammonia water, from time to time during the evaporation, aids in getting a completely soluble product.

Evaporate and scale as in making ferric citrate, only spread the thin syrup very thinly on glass. If spread even as thin as ferric citrate, opaque scales are formed. If very thin, beautiful pearly scales result.

MAKE LEAD PLASTER, U. S. P.

Recipe.—Soap, dried, and in coarse powder..... 10 Gm.
Lead acetate..... 6 Gm.
Water, a sufficient quantity.

Dissolve the soap in 35 Cc. of hot water and strain the solution. Dissolve the lead acetate in 25 Cc. of hot water, and at once filter the solution into the warm soap solution, stirring constantly.

When the precipitate has subsided, decant the liquid, and wash the precipitate thoroughly with hot water. Transfer the mass to a warm slab, kneading it thoroughly to free it from water. Finally, roll the plaster into cylindric forms and wrap them in paraffined paper.

Chemical Explanation.—Lead plaster of the present pharmacopœia, while approximately of the same composition as lead plaster U. S. P. 1890 (lead oleate), is made by the much simpler expedient of precipitation by mixing solutions of Castile soap (sodium oleate) with a solution of lead acetate, the reaction being *sodium oleate plus lead acetate equals lead oleate plus sodium acetate*.

Write out the equation showing this reaction.

As neither Castile soap nor lead plaster are definite chemicals, no attempt will be made to calculate quantities by molecular weights.

Remarks on Process.—*Dissolve the soap* in pint flask, and strain into pint evaporating dish.

Dissolve the lead acetate in another pint flask (or same one, carefully washed), and filter into soap solution in dish.

Knead the mass on pill-tile.

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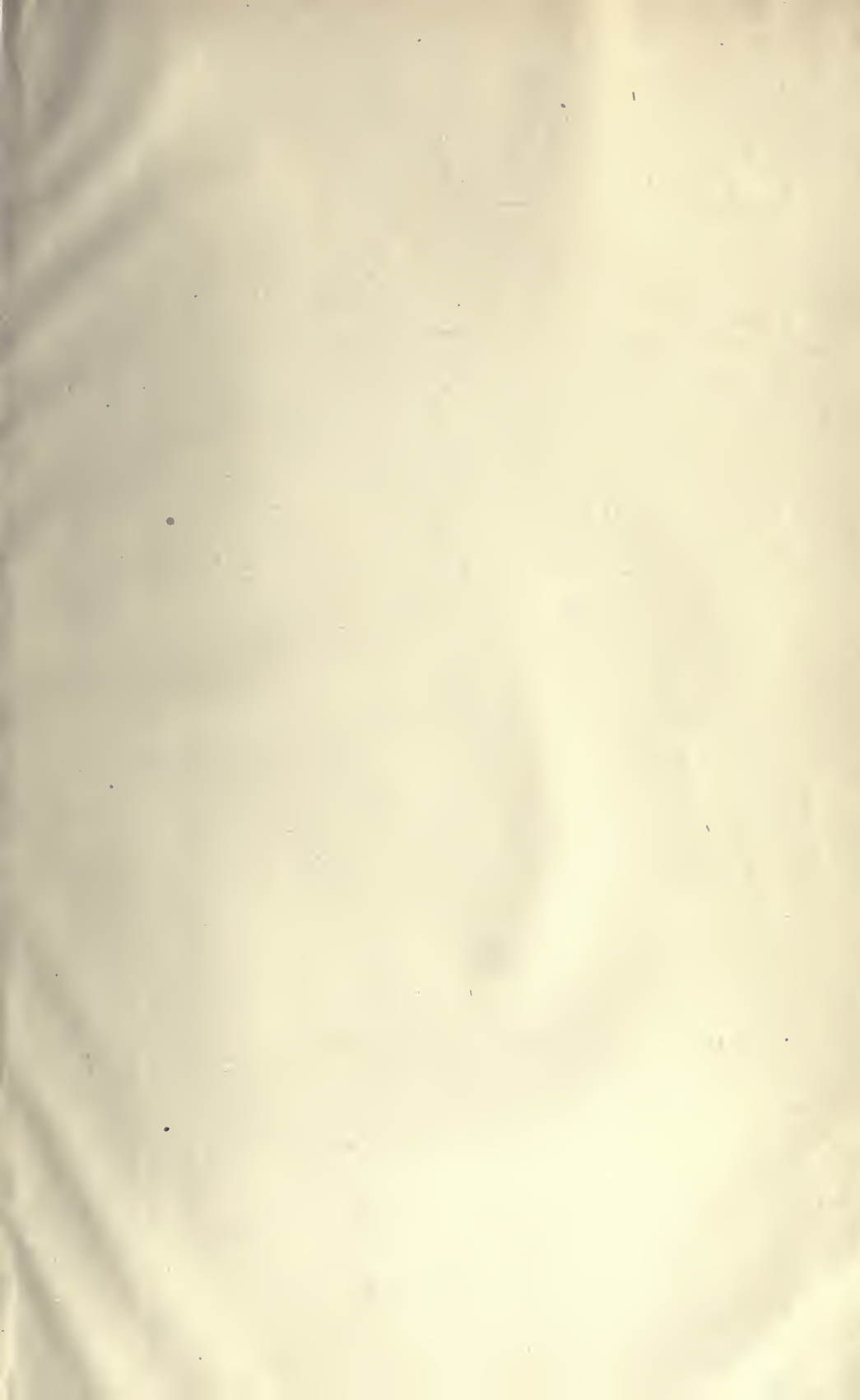
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